A STUDY OF CHERT AND SHALE GRAVEL IN CONCRETE

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Joint Highway Research Project

PURDUE UNIVERSITY
LAFAYETTE INDIANA

Final Report

A STUDY OF CHERT AND SHALE GRAVEL IN CONCRETE

TO: K. B. Woods, Director
Joint Highway Research Project

September 13, 1960

FROM: H. L. Michael, Assistant Director
Joint Highway Research Project

File: 5-9-6
Project: C-36-42F

Attached is a final report entitled, "A Study of Chert and Shale Gravel in Concrete". The report was prepared by Mr. R. L. Schuster under the direction of Professor J. F. McLaughlin. Mr. Schuster also used this report as his thesis for the Ph. D. degree.

The report is presented to the Board for the record.

Respectfully submitted,

H. L. Michael /jm

Harold L. Michael, Secretary

HLM:JFM:kr

Attachment

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Final Report

A STUDY OF CHERT AND SHALE GRAVEL IN CONCRETE

by

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Joint Highway Research Project
File: 5-9-6
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Purdue University
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TABLE OF CONTENTS

TABLE OF CONTENTS (continued)

TABLE OF CONTENTS (continued)

TABLE OF CONTENTS (continued)

LIST OF TABLES

LIST OF TABLES (continued)

LIST OF TABLES (continued)

LIST OF ILLUSTRATIONS

LIST OF ILLUSTRATIONS (continued)

ABSTRACT

Schuster, Robert Lee. Ph.D., Purdue University, January, 1961.

A Study of Chert and Shale Gravel in Concrete.

Major Professor: John F. McLaughlin.

Certain chert and shale gravels have long been recognized as harmful when included in portland-cement concrete exposed to freezing and thawing. Many organizations have specifications limiting percentages of these materials in concrete aggregates, but few of these specifications distinguish between types of chert and shale from different geographical areas nor do they always take into account the basic physical properties of these materials.

In this study, pore characteristics, mineralogy, texture, and structure were determined for cherts and shales from nine Indiana glacial gravel deposits by means of microscopic petrography, x-ray diffraction, and the common specific gravity and absorption techniques. Blends of two, four, six and ten per cent of chert or shale from each source were made with a standard durable crushed limestone coarse aggregate, and these blends were used in 3- by 4- by 16-inch air-entrained concrete beams subjected to up to 300 cycles of freezing-and-thawing. A measure of the amount of deep-seated deterioration of the beams was provided by durability factors calculated from the results of non-destructive sonic testing of the beams at intervals

during freeze-thaw testing. Severity of surface deterioration was also evaluated. The influence of the basic properties of the chert and shale gravels on the results of the freeze-thaw tests was then determined. On the basis of the results of these tests, study was made of the existing specifications on cherts and shales to determine whether the specifications realistically categorize these materials.

Despite significant differences in their mineralogies, no difference was noted in the freeze-thaw durabilities of the various chert samples. For all the cherts, significant deep-seated and surface deterioration occurred only in beams containing six to ten per cent of material with a bulk specific gravity (saturated surface-dry basis) of less than 2.45.

Although the basic properties of the shales varied even more widely than those of the cherts, none of the shales caused deep-seated failure of the concrete. However, the most porous shales caused "popout" damage which was especially severe at the six to ten per cent levels.

INTRODUCTION

The presence of certain rocks and minerals in aggregates may greatly impair the quality of concrete made with these aggregates. The term "deleterious substances" or "deleterious constituents" has become a common one for describing this class of materials.

Deleterious substances are ones which have an adverse effect on the concrete. These substances may be categorized on the basis of the nature of their harmful effects (6)[1]. The most harmful class of deleterious aggregates consists of those which under certain conditions will expand disruptively in the concrete. The most common examples of this class are porous cherts, limonite concretions, well-indurated shales, and limestones containing expansive clays. Such materials, when present in concrete and frozen in a saturated condition or, in some cases, when merely exposed to water, increase in volume and develop sufficient pressure to cause deep-seated integration of the concrete.

In another class of deleterious substances the aggregate particles do not undergo volume changes which tend to disrupt the surrounding concrete. Instead, when frozen in a saturated condition, they break into numerous smaller pieces due to the inherent structural

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[1] Numbers in parentheses refer to references listed in the Bibliography.

weaknesses of the particles themselves without exerting much pressure on the surrounding concrete. Examples of such structurally weak deleterious substances are clay lumps, ocher, poorly indurated shales, and soft sandstones. Depending on the quantity of these materials used in the concrete, deterioration may be general or, more often, may be evidenced primarily by surface pitting or "popouts."

Other materials which are commonly classified as deleterious are organic impurities and lightweight pieces, including coal and lignite. Organic impurities generally retard the setting of cement and reduce concrete strength, particularly at early ages. Finely divided coal or lignite in sufficient quantity will also retard hardening.

This study deals primarily with cherts[1] and shales[2] because these two rock types are probably the most abundant of the deleterious constituents of Indiana's gravel aggregates. There is still much to be learned about the physical properties of these two materials and their effects on the durability of concrete.

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[1] Chert may be defined as a dense cryptocrystalline sedimentary rock, composed of chalcedony (microcrystalline fibrous silica and microfibrous amorphous silica or opal) and cryptocrystalline quartz (38). It has a tough splintery to conchoidal fracture. It is commonly white, gray, or blue-gray, but may be brown, black, green, blue, pink, red, or yellow. Flint is a term widely used both as a synonym for chert and as a variety of chert. Tarr (61) states that flint is identical with chert and recommends that flint be dropped from geologic usage. Although the term flint antedates the term chert, present-day usage favors the latter as the proper designation of the materials to which both terms have been applied (38).

[2] Shale may be defined as a laminated sediment in which the constituent particles are predominantly of clay size (17). Shales consist of lithified muds and clays that are fissile and break along planes parallel to the original bedding. A typical shale is so fine-grained as to appear homogeneous to the unaided eye, is easily scratched, and has a smooth feel (56).

REVIEW OF LITERATURE

This literature review deals with the problem of deleterious substances in concrete aggregates, and in keeping with the theme of this study, it is especially concerned with the problem of cherts and shales in concrete aggregates.

The review is divided into two parts. The first of these deals with the problem of deterioration of concrete due to chert aggregates. In the second part, studies of the effects of shale aggregates on concrete deterioration are reviewed. All available references in the field are not included in this review; rather it is a restricted coverage that attempts to give a concise version of what has been accomplished in studies of chert and shale durabilities in the past.

Research on the Relationship Between Chert Aggregates and Concrete Deterioration

It has long been recognized that certain types of aggregates have harmful effects on the concrete in which they are used. It was not until the 1920's, however, that much research was begun in this country to quantify the effects of the deleterious materials, and thus to determine the quantities of these materials that could be included in concrete aggregates. During the construction season of 1923, Reagel (43) noticed a peculiar surface effect on the concrete pavements constructed in certain localities in Missouri. Investigation

showed that portions of the surface averaging 1 to 1 1/2 inches in diameter were cracking loose from the pavement and could be pried out or were later displaced by traffic, leaving a hole with sloping sides from 1/2 inch to 1 inch in depth. After removal of a large number of these "popouts", it was discovered that the piece of pavement loosened always contained a large fragment of chert aggregate at the bottom and the remainder of the piece of chert was left forming the bottom of the hole in the pavement. It was evident that the force acting to raise the popout occurred in all cases through the chert aggregate. The action never occurred in connection with pieces of limestone, of which the greatest part of the coarse aggregate in the pavement consisted, but always just above or through a piece of chert.

Reagel then conducted an investigation which showed that these popouts were due to frost action on the pieces of chert. He subjected aggregates that were high in chert content to simple freezing-and-thawing tests in the laboratory. These tests, which consisted of five cycles of alternate freezing and thawing, were conducted on both the loose aggregate and on concrete beams in which the aggregate was incorporated. The tests showed that the loose chert was seriously affected by alternate freezing and thawing, and often disintegrated completely. The test beams showed a definite loss of strength; and, in cases where large percentages of chert were included in the aggregate, these beams often disintegrated to the point where they could not support their own weight.

Reagel noted that the five cycles of freezing and thawing which he used in these tests were considerably fewer than could ordinarily

be expected in an average winter season in Missouri. Since five cycles of freezing and thawing often produced severe disintegration of concrete made from the chert aggregate, he concluded that chert should be limited to the smallest percentage possible in aggregates to be used in the production of concrete.

In 1928, Scholer (52) stated that, "The use of unsound aggregate produces unsound concrete, the resistance of the mortar to disintegration being only slightly effective in protecting the aggregate." In order to determine which aggregates produced potentially non-durable concrete, he developed a method that tested the resistance to freezing and thawing of concrete cylinders made from the aggregates in question.

Using freezing-and-thawing tests of concrete, Scholer (51) continued his research on various aggregates that were suspected of containing substances potentially harmful to concrete. In the Kansas aggregates which he tested, he found that the most common and most destructive deleterious substances were absorptive chert in gravel and flinty concretions in limestone.

Runner (49), in 1937, was one of the first seriously to apply petrography to the study of deleterious substances in aggregates. He found that it was possible to determine the probable durability of aggregates by means of studies using the petrographic microscope. He made thin sections of aggregate pieces and studied them under the microscope. He was able to identify the harmful types, such as certain cherts, on the basis of porosity, texture, and mineral compo-sition.

In 1938, Litehiser (30) published the results of investigations

of the effects of Ohio aggregates on concrete durability. Using the freezing and thawing test developed by Scholer (52) and the sodium sulfate soundness test, he found that shale, limonite, ocher, hematite, iron pyrite, and some varieties of chert had detrimental effects on concrete in which they were used. He noted that not all varieties of chert were deleterious, but did not suggest a means for differentiating between deleterious and non-deleterious ones.

Also in 1938, Gibson (14) presented data from an extensive performance study of concrete structures in which were incorporated sand and gravel from the Arkansas, Platte, and Kaw Rivers. He found that the predominantly siliceous sands and gravels of the Platte River had caused considerable distress when used in pavements in Missouri, Kansas, and Nebraska.

In 1939, Cantrill and Campbell (9) published the results of a concrete pavement condition survey they had conducted in Kentucky. Analysis of their data showed that serious failures of concrete pavements throughout the western part of Kentucky were due to the use of chert gravels obtained from the Tennessee and Cumberland Rivers in the western part of the state. Pavements in which these chert gravels were used often began to disintegrate within one year after construction.

The results of this survey led Cantrill and Campbell to a laboratory study of the western Kentucky cherts. They found these cherts to be extremely porous, highly absorptive, and possessed of a low specific gravity. The chert gravels passed all the standard laboratory tests for abrasion and soundness which were used at that time. These were: (a) the Los Angeles Abrasion Test, (b) the Deval Abrasion Test,

and (c) the Sodium Sulfate Soundness Test. Also strength tests on chert-gravel concrete field specimens showed values comparable to those for specimens made from Ohio River gravel or crushed limestone with good service records. However, when the chert aggregate was incorporated in concrete beams and subjected to 40 cycles of freezing and thawing in water, a definite reduction in flexural strength was noted. It was thus concluded that freezing and thawing was the cause of the disintegration of these lightweight Kentucky cherts when used in concrete pavements.

In 1940, Wuerpel and Rexford (74) published the results of their investigation of the possibility of separating durable and non-durable varieties of chert in concrete coarse aggregate by some means more precise than visual examination and more practical than microscopic analysis. In their paper, they included a symposium of related comments by other investigators. Included in this symposium are the following significant comments from the Corps of Engineers' Rock Island investigation (63):

1. "Heating and cooling of chert had absolutely no effect. This test was initiated to allay some suppositions that chert caused "popouts" in summertime due to heat of the sun followed by a cooling rain or vice-versa."

2. "These results [freezing and thawing of paraffin-coated chert in mortar specimens] indicate that popouts occur only from freezing of water absorbed by the chert. This hypothesis is further verified by a comparison of the absorption and specific gravity of each type of chert with its reaction to freezing and thawing. It seems a general rule that a chert stone with an absorption greater than 3 per cent or an apparent specific gravity less than 2.50 can be classified as harmful material."

Wuerpel and Rexford collected samples of cherty gravel from ten areas in the southern,central, and eastern portions of the United States. These were separated into four bulk-specific-gravity groups by heavy-liquid flotation using bromoform (specific gravity 2.86) and mono-bromo-benzene (specific gravity 1.46). The groups used had specific gravities of 2.50 plus, 2.40 to 2.50, 2.30 to 2.40, and 2.30 minus. Material in each group was analyzed microscopically and tested physically for absorptive capacity, resistance to frost action, and resistance to a magnesium sulfate soundness test. The results of these tests showed a definite increase in absorption and decrease in soundness with lower bulk specific gravity of the samples. These trends were present for all groups lower in bulk specific gravity than 2.50, but were especially noticeable in the pebbles having a bulk specific gravity of less than 2.40.

These results compared favorably with the results of a performance survey of the exposed concrete structures in the areas from which the gravels had been obtained. A group of 100 roughly conical popouts, each having a piece of disrupted chert at the apex, was collected from representative structures. In every case, the piece of chert had an absorption greater than four per cent and a bulk specific gravity less than 2.40.

On the basis of this investigation, Wuerpel and Rexford concluded that the flotation method of specific gravity separation is the most practical method of separating durable from non-durable cherts. They recommended that the flotation test be used as a field test for the separation of a majority of the non-durable chert in concrete aggregate. In addition, they developed a flotation field kit to be

used for this purpose.

In their discussion of the preceding study by Wuerpel and Rexford, Reagel and Willis (45) considerably contributed to the knowledge of the durability characteristics of chert. Their research was conducted on a Missouri chert-rich gravel coarse aggregate with a poor service record in concrete pavements. This aggregate, as produced for concrete pavement, had an average bulk specific gravity of 2.51 and, in the stream-wet condition, 3.8 per cent absorbed moisture. By means of a technique similar to that used by Wuerpel and Rexford (74), the saturated aggregate was separated into three fractions of different bulk specific gravities, namely, less than 2.4, 2.4 to 2.5, and over 2.5. These three coarse aggregate gravity fractions were then incorporated in 3 1/2- by 4 1/2- by 16-inch beams which, after a 28-day curing period, were subjected to consecutive cycles of freezing in air and thawing in water. After 1, 3, 5, 7, 9, and 10 cycles, the dynamic modulus of elasticity and the specimen length were determined for each of the beams.

The results of these tests showed that, for the particular aggregate employed, resistance to the freezing-and-thawing cycle used was much greater for the concrete containing the aggregate fraction of greatest bulk specific gravity than for that containing aggregate fractions of lower gravity. The results also indicated that removal of the low-gravity aggregate fractions (less than 2.50) and use of only the highest fractions (over 2.50) produced concrete that was more resistant than that in which the unseparated stream-run gravel was used.

One of the principal points brought out by Reagel's and Willis' tests was that concrete made from even the highest gravity fraction (all particles were over 2.5 and an average bulk specific gravity of 2.58) of this chert-rich aggregate having a poor service record showed considerably less resistance to freezing and thawing than that of a non-chert aggregate with a good service record.

In the early 1940's, White and Peyton (70), conducted a performance survey of 1,170 miles of concrete pavement in Kansas. The purpose of the survey was to investigate the relationship between certain classes of failure (mainly D-line cracking) and the coarse aggregate incorporated in the concrete. The coarse aggregate consisted of five general types: (a) crushed limestone, (b) chert gravels, sometimes crushed, (c) crushed flint, (d) flint chat, and (e) sand and gravel (mixed aggregate). Predominantly poor records were shown by pavements constructed with Joplin flint, Bazaar gravel (a chert gravel), and the Moline and Kansas City limestones. The Joplin and Bazaar materials undoubtedly failed due to their mineralogic contents. Although no mineralogic analyses were given for the Moline and Kansas City limestones, it is quite possible that the failures of these aggregates were also due to the presence of chert or flint as impurities.

In 1941, Reagel and Gotham (44) published the results of their performance survey of some 450 miles of concrete pavements in Missouri. They were mainly interested in transverse cracking and blowups in the pavements. They concluded that pavements built with coarse aggregate produced from the chert deposits of Missouri tend to develop more transverse cracks and blowups than comparable pavements built with

crushed limestone aggregate. Comparisons showed that at five years of age pavements built with limestone had developed 10.5 transverse cracks per 1,000 feet or an average crack interval of 95.2 feet, while comparable chert aggregate pavements had 26.1 cracks per 1,000 feet or a crack interval of only 28.3 feet. This trend persisted for the older pavements also. At 14 years of age the limestone pavements had an average crack interval of 61.3 feet (16.3 cracks per 1,000 feet), whereas the chert pavements had cracks at an average interval of 26.2 feet (38.1 cracks per 1,000 feet). These data show that, on the average, unreinforced pavements without joints built with chart aggregates developed in 14 years over twice as many cracks as occurred at the same age in comparable pavements built with limestone aggregates.

Comparison of blowups in pavements built with the two types of aggregates showed an average of one blowup every 4,800 feet for unreinforced pavements made from crushed limestone aggregates, and one blowup for every 1,055 feet of pavement produced from chart aggregates (1.1 blowups per mile versus 5.0 blowups per mile).

About this time the deleterious substances in Indiana's aggregates also began to be exposed to comprehensive study. In 1940, Sweet (57), and, in 1942, Sweet and Woods (60) published the results of thorough investigations of chert in Indiana's aggregates. They identified the chert in samples of aggregate and studied it by means of chemical and microscopic analysis, mineralogical examination, absorption tests, and freezing-and-thawing durability tests. They recognized the fact that all cherts are not unsound, and attempted to find a

means of differentiating between durable and non-durable varieties. They considered unsound aggregates to be those which are unable to resist excessively large or permanent changes in volume when subjected to destructive agencies, particularly freezing and thawing, heating and cooling, or wetting and drying.

The first step in this study was the collection of samples from quarries and gravel deposits. Approximately four-thousand pounds of chert were secured from the six State Highway Districts in Indiana, and from sources in Illinois, Kentucky, Michigan, Missouri, Ohio, and Tennessee. The Indiana ledge rock samples were obtained from 29 quarries and highway cuts, and the gravel samples from 31 gravel deposits in all parts of Indiana.

It was decided to test the performance and properties of the quarry samples first, because the properties of the individual pieces in each sample were reasonably uniform. Ledge cherts were obtained from quarry faces and identified geologically. A record was made of the macroscopic character of each sample, including color, luster, texture, and nature of fracture. Each sample was subjected to performance and identification tests.

Performance was determined by embedding vacuum-saturated chert pebbles in mortar cubes and subjecting the cubes to alternate cycles of freezing and thawing. The blocks were frozen in water for 21 hours at minus 10°F., and then were immersed in water at 75°F. to 80°F. for three hours. This procedure was repeated until 40 cycles had been reached. At intervals the cubes were examined for signs of cracking. On the basis of these tests, the cherts were divided into two groups: (a) those which disrupted the cubes 51 to 100 per cent of the time

in less than 40 cycles; and (b) those which disrupted the cubes 0 to 50 per cent of the time in 40 cycles.

Identification tests of the quarry cherts consisted of bulk specific gravity, absorption, degree of saturation, dye penetration, unconfined freezing and thawing, chemical analyses, and microscopic examination of thin sections. The most useful results were those from the specific gravity and absorption tests. The cherts in group A (those which disrupted the cubes 51 to 100 per cent of the time in less than 40 cycles) had an average bulk specific gravity, saturated surface-dry, of 2.40 and a maximum of 2.46. Their average absorption was 5.36 per cent and the minimum absorption for group A cherts was 3.91 per cent.

Samples in group B (those cherts which disrupted the cubes 0 to 50 per cent of the time in 40 cycles) averaged 2.58 in bulk specific gravity, saturated surface-dry, with a minimum of 2.48. Absorption of cherts in this group averaged 1.88 per cent with a maximum of 3.02 per cent.

The same general performance test procedure employed on the quarry cherts was used to determine the durability of the gravel cherts. Pieces 3/4 inch to 1 inch in size were picked at random from samples of gravel chert from Indiana and other states. These were evacuated for one hour, saturated, and immersed for 24 hours. They were then embedded in 2-inch mortar cubes. The mortar cubes were moist-cured for seven days and subjected to the freezing-and-thawing test. The gravel chert specimens that failed in this test were removed from the broken mortar and subjected to the following identi-

fication tests: color, texture, bulk specific gravity by the flotation procedure, dye penetration, apparent specific gravity, absorption, degree of saturation, and microscopic analysis. The non-failing specimens were removed from the freezing and thawing test at the end of 40 or 160 cycles, broken from the cubes, and then analyzed, using the same identification tests as were used with the failures.

A modification of the flotation method developed by Wuerpel and Rexford (74) for determining the bulk specific gravity of gravel particles was used in separating the chert samples into different fractions on the basis of their specific gravity. Carbon tetrachloride, specific gravity 1.58, and acetylene tetrabromide, specific gravity 2.97, were mixed together to give liquids with specific gravities of 2.60, 2.55, 2.50, 2.45, 2.40, 2.35, and 2.30. A gravel specimen that had been broken out of the mortar cubes was immersed in water. After it had soaked for 24 hours, it was surface-dried and placed in the heaviest liquid (2.60). If it sank in this liquid, it was removed, and the specific gravity was recorded as 2.60 plus. If it floated on the 2.60 liquid, it was removed and placed in the 2.55 liquid. This was repeated for each piece until it sank in one of the liquids. If a piece floated in the lightest liquid (2.30), its specific gravity was recorded as 2.30 minus.

In this way it was possible to obtain a correlation between the bulk specific gravity, saturated surface-dry, of a piece of chert, and its performance in the freezing-and-thawing test. The results of these studies showed that the average bulk specific gravity, saturated surface-dry, of unsound chert was lower than that of durable

chert. They indicated that an upper limit of 2.30 detected entirely unsatisfactory material; 2.45 detected almost all the very harmful types, and included little durable material. A limit of 2.50 included almost all the non-durable material and also a somewhat larger amount of relatively durable particles than did the 2.45 limit.

Even though Sweet and Woods found in these tests that no sharp line could be drawn between entirely sound and entirely unsound chert on the basis of specific gravity, they were able to set up the following table as a relative measure of the probable performance of Indiana cherts in concrete based on bulk, saturated surface-dry, specific gravity:

Below 2.30	-	Unsatisfactory
2.30 - 2.45	-	Poor
2.45 - 2.55	-	Fair
2.55 - 2.60	-	Good
Above 2.60	-	Excellent

It should be noted that in this chart proposed by Sweet and Woods, the break between "good" and "bad" cherts was found to occur at a specific gravity of 2.45. This same specific gravity is in use today by the Indiana Highway Department as the level of separation between durable and non-durable cherts.

The absorption of a piece of aggregate is ordinarily directly related to the specific gravity of the piece since, in most cases, the absorption of a material is dependent upon its porosity (27). Porosity is, in turn, directly related to the bulk specific gravity of the material. Therefore, the relative absorption of an aggregate particle may be used as an indication of the durability of the particle in the

same way as bulk specific gravity.

A simple means for measuring the relative absorptivity of aggregates was used by Sweet and Woods. They selected pebbles about 1 1/2 inches in diameter, partially immersed them in a one-per cent solution of water-soluble eosine dye for a given period of time, and then measured the depth of penetration of the dye. They found that the greater the penetration of the dye, the lower the durability of the chert. On the basis of these tests, they proposed the following table of one-hour dye penetration depths to be used for predicting the relative durability of Indiana gravel cherts:

0.25" or more			-	Unsatisfactory
0.20"	-	0.24"	-	Poor
0.10"	-	0.19"	-	Fair
0.05"	-	0.09"	-	Good
0	-	0.04"	-	Excellent

In the early 1940's, a comprehensive survey was made of most of the portland-cement concrete pavements which were in use in Indiana. The most important result of this survey was the correlation found between the source of natural aggregate used in the portland-cement concrete and the performance of the completed pavements. This survey, the results of which were published in 1946 by Woods, Sweet, and Shelburne (72), included studies of 3,300 miles of pavement, or about 78 per cent of all the rigid pavements constructed in Indiana between 1921 and 1943. This mileage contained a total of 725 projects, with cements from 17 sources, fine aggregates from 138 sources, and coarse aggregates from 155 sources. Considerable attention was given in this survey to general deterioration and to blowups.

From the results of the study a correlation was established for

Indiana pavements between certain sources of coarse aggregate incorporated in the concrete mix and the susceptibility of the resulting pavement to blowups. It was found that 284 miles of pavement (only 10.8 per cent of the total mileage studied) constructed from five sources of supply, contained 1,168 blowups (49 per cent of the total blowups). The crushed stone from one of these five sources was used to construct 97.1 miles of pavement (only 3.7 per cent of the total surveyed) which was found, in this survey, to contain 707 blowups (29.4 per cent of the total blowups). This correlation of blowups with sources of coarse aggregate resulted in the previously unexpected conclusion that expansion joints were not needed in Indiana concrete pavements.

Although Woods, Sweet, and Shelburne did not perform mineralogical analyses on their aggregate samples, later investigators (4, 8, 54) did analyze gravels from the sources which had resulted in aggregates producing concrete pavements especially susceptible to blowups. These analyses showed that, in most cases, the gravel aggregates with poor performance records contained excessive amounts of chert.

In the early 1940's, it was first suggested that air entrainment could be used to improve the durability of concrete susceptible to freezing-and-thawing failure due to content of unsound coarse aggregates such as porous cherts. Considerable research was begun at that time in an attempt to verify this hypothesis.

In 1943, Axon, Willis, and Reagel (2) fabricated air-entrained concrete test beams from four different Missouri coarse aggregates: two crushed limestones, and two chert-rich river gravels. The two limestone aggregates had good service records; one of the chert-rich

gravels had a fair service record, while the other had produced only concretes with poor durability. Each of these coarse aggregates was used, in a saturated condition, in three separate batches. One of these batches was made with plain portland cement and contained about one-per cent air. The other two batches contained a blend of plain cement and cement ground with 0.1 per cent vinsol resin to give entrained air contents of 4 and 7 per cent for the respective batches. After curing, the beams from these batches were subjected to the freezing-and-thawing test, and their flexural strengths were measured.

Results for both limestone concretes containing entrained air showed a definite improvement in durability as measured by the freezing-and-thawing test. However, for concrete made with the chert-rich aggregate with a fair service record, the use of entrained air resulted in only a slight increase in durability. In the case of the chert-rich aggregate with a poor service record, the use of entrained air resulted in no appreciable improvement in durability. Axon, Willis, and Reagel concluded that there is only a slight chance that air entrainment will appreciably reduce the rate of disintegration resulting from freezing and thawing of concrete containing unsound aggregates.

In 1944, Lindsay (29) investigated the relative durability of air-entrained portland-cement concrete and regular portland-cement concrete made with chert-rich aggregates. The aggregates used were stream-saturated gravels. Little or no improvement in durability resulted from the use of the air-entrained portland cement.

In the same year, Reagel (42) published the results of further experiments of this type. The tests conducted by Reagel were similar

to those used by Axon, Willis, and Reagel (2) in 1943. As in these earlier tests, he investigated the effects of air entrainment on concrete made from two saturated chert-rich gravels and two crushed limestone aggregates as measured by resistance to laboratory freezing and thawing. His results showed that entrained air improved the durability of concrete containing either mediocre or good limestone aggregates, but caused no appreciable improvement in the durability of concrete containing chert-rich aggregates.

Also in 1944, S. Walker (66) published the results of freeze-thaw studies of concrete made with gravels sampled from 70 coarse aggregate deposits throughout the United States. A freezing-and-thawing cycle of 17 hours in air at 0°F. and 7 hours in water at 40°F. was used. The effect of freezing and thawing was measured by noting change in the dynamic modulus of elasticity of each specimen. The test was continued on each beam until the modulus of elasticity was reduced to about 50 per cent of its original value, and then strength in flexure was determined. An empirical durability factor was determined for each specimen on the basis of the results of the freeze-thaw testing. It was found that several glacial gravels from the middle west, containing various amounts of white porous chert, had considerably lower than average durability factors.

In 1947, Bugg (8) investigated the effects of air entrainment on concrete containing Indiana aggregates. He used the same techniques as previous investigators except that some batches were made using aggregates which were vacuum saturated while for other batches the aggregates were merely immersed in tap water at room temperature for

24 hours. Bugg worked with four different aggregates: two crushed limestones and two chert-rich gravels. One of the limestone aggregates had a good field performance record while the other had only a fair record. One of the chert-rich gravels contained only 9 per cent chert and had a fair field performance record. The other chert-rich gravel contained 43 per cent chert and had a very poor field performance record.

Concrete beams made from these aggregates and from either regular or air-entrained portland cement were subjected to the freezing-and-thawing test. From the results of this study Bugg concluded that under the conditions of the freezing-and-thawing test and with the materials used, air-entrained concrete showed slight to considerably greater durability in every condition investigated than did regular cement concrete. However, it should be noted that the greatest improvement was shown by the immersed aggregates. Later studies by Sweet (58) have shown that 24-hour immersion does not approximate the high degree of saturation that many river gravels have at the time of their production for aggregate. Sweet also found that freezing and thawing of laboratory-fabricated concrete beams produced results that were in accord with the field performance of the materials used when the aggregate was incorporated in the concrete in a moisture condition corresponding to this field saturation. At lower degrees of saturation, aggregates with poor service records were highly resistant to laboratory freezing-and-thawing tests. Sweet's research indicates, therefore, that the results of the tests which Bugg conducted on concrete made from immersed aggregates may not be truly indicative of the situation existing in the pavement.

For the saturated aggregates, Bugg's results were actually similar to those obtained in earlier studies by other investigators. He found that air entrainment improved the durability of concrete made from the limestone aggregates and from the chert-rich aggregate with a fair performance record. The improvement shown by the saturated aggregate containing 43 per cent chert, however, was not appreciable except where percentages of entrained air were used which were large enough to seriously affect the strength of the concrete.

Also in 1947, Soon (54) carried out a series of tests on coarse aggregate from eight sources of supply commonly used in concrete pavements in Indiana in which he attempted to determine the relationship between field and laboratory performance of the aggregates. He divided the samples into bulk specific gravity ranges by means of the heavy-liquid flotation process, and pieces from each range were embedded in 2-inch mortar cubes and subjected to freezing-and-thawing action. The cubes were placed in pans containing about an inch of water and frozen for 21 hours at 0°F. to 10°F. They were then immersed in water at 75°F. to 80°F. for three hours. This procedure was repeated until failure of the cubes or until a given number of cycles was attained. In the failure of a cube, the usual process was the appearance of cracks which became progressively worse with each cycle of freezing and thawing until the fractured cube could be pulled apart by hand using a moderate amount of force.

On the basis of the results of these tests, Soon concluded that Indiana aggregates are increasingly durable in the following order: (a) soft particles, (b) cherts, (c) limestones below 2.50 in bulk

specific gravity, (d) sandstones, (e) shales, (f) limestones above 2.50 specific gravity.

Lewis and Venters (28) furthered the study of the deleterious constituents of Indiana aggregates by separating large samples of gravels into fractions having different specific gravity ranges, and testing these gravels for absorption, degree of saturation, and durability as indicated by the freezing-and-thawing durability test. The freezing-and-thawing test was conducted on 3- by 4- by 16-inch concrete beams in which different aggregate fractions were used. The test consisted of freezing in air at minus 15°F. to minus 20°F. and thawing in running tap water at 55°F. to 60°F. One cycle per day was obtained, with 16 hours freezing and 8 hours thawing. Periodic determinations of the dynamic modulus of elasticity were made to measure the deterioration of each specimen.

The results of these tests showed that aggregates with low specific gravities were characterized by high absorptions, high degrees of saturation, and poor durability in concrete subjected to freezing and thawing. The deleterious substances in the low-specific gravity fractions consisted mainly of cherts and sandstones with lesser amounts of igneous, calcareous sedimentary, and metamorphic rocks. The poor freeze-thaw durability of concrete made with low-specific gravity aggregates was of great importance because Lewis and Venters found that those gravels in this study which had low specific gravities also had poor field records.

As a result of their investigation, Lewis and Venters suggested that increased durability of concrete produced in actual construction

could be obtained by the use of field heavy-media-separation processes.

They stated that separation at 2.40 specific gravity would improve the durability of poor aggregates considerably and separation at 2.50 would result in aggregate with good durability.

R. Walker and McLaughlin (65) experimented further with combinations of Indiana aggregates. They used heavy-liquid separation to obtain from gravels various fractions with different minimum specific gravities. They then used these fractions, alone or in combination with good-quality crushed stone, in concrete which was tested for durability in freezing and thawing.

The gravels which were studied all had high chert contents. They contained from 10 to 70 per cent chert. Although the specific gravity separation removed other low-specific gravity materials also, most of the material removed was chert.

Walker and McLaughlin found that removal of the low-specific gravity fractions from the gravel aggregates resulted in a concrete of higher durability than that made from the original, unseparated aggregate. Also, concrete made with crushed stone-gravel combinations, where the gravel used had poor service records, was made more durable with the heavy-liquid separation. They also found that the durability of concrete made with gravel aggregates alone compared favorably with field performance of the aggregates, thus indicating the validity of results of the freezing-and-thawing test as a relative indication of the durability of concrete.

In 1956, Legg (26) published the results of laboratory freeze-thaw tests of the major sources of Michigan gravel aggregates. He

evaluated coarse aggregate durability following a rigidly standardized procedure wherein the aggregates were graded, and then placed in air-entrained concrete in a vacuum-saturated condition. ASTM Designation: C291-52T, Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water, was used for the test. Failure was considered to have occurred when the relative dynamic modulus of elasticity of 3- by 4- by 16-inch beams had decreased by 30 per cent. Durability factors based on 300 cycles of freezing and thawing were used to describe the behavior of the aggregates.

On the basis of the laboratory freeze-thaw studies, Legg found that, in general, those gravels containing considerable percentages of deleterious materials such as chert, shale, clay ironstone (limonite concretions), and sandstone had low durability factors. In special studies utilizing heavy-media separation of chert-rich gravels, he found chert with high bulk specific gravity to be far less harmful than chert having a low specific gravity.

On the basis of the research previously described, other research, and actual experience with chert-rich gravels in service, the American Society for Testing Materials and several state highway departments, including those in Indiana, Michigan, Ohio, and Illinois, now have specifications that control the amount of chert allowed in concrete aggregate (53). For instance the Indiana Highway Department's 1957 standard specifications (55) state that coarse aggregates shall contain a maximum of 3.0 per cent of chert with a bulk specific gravity (saturated surface-dry basis) of less than 2.45.

Research on the Relationship Between Shale Aggregates and Concrete Deterioration

It has long been suspected that shales have a part in the deterioration of concrete in which they are used as aggregate. In 1927, Lang (25) showed a direct relationship between percentage of shale in gravel used as concrete aggregate and loss of compressive strength for the resulting concrete. This loss in strength is generally considered to be due to the inherent weakness of the shale particles themselves. The effect of shale on the freeze-thaw durability of concrete is not so clearly defined as its effect on the strength of concrete, however. For quantities of shale up to five per cent, Lang found about two per cent reduction of strength for each one per cent of shale when the concrete was subjected to eleven cycles of freezing and thawing. As a result of his studies, Lang concluded that for pavement concrete the only harmful effect of shale consists of surface deterioration of the concrete.

In a later report, Lang (24) emphasized the difficulty of evaluating the deleterious effects of shale. He pointed out that the properties of shales vary considerably from one area to another, and that the freeze-thaw durability of one shale may differ considerably from that of another shale. He also noted that shales grade into other rock types such as slates or siltstones to further confuse the situation.

The Kentucky State Highway Department also carried out some early studies of the effects of shales on concrete durability (19). They studied the effects of sodium sulfate solutions and of natural weather-

ing on concrete made with varying percentages of a "low grade blue shale" coarse aggregate and concluded the blue shale should be limited in use in concrete to less than five per cent of the coarse aggregate.

The Tennessee State Highway Department carried out a study of the effect of shale in coarse aggregate on the strength of concrete and on its resistance to freezing and thawing. S. Walker and Proudley (67) note that in this investigation the concrete specimens were made in two groups, one of which was subjected to normal curing conditions and the other to freezing-and-thawing conditions. The report of this study states that the inclusion of five and ten per cent of shale in the coarse aggregate had no effect on the durability of concrete subjected to 45 cycles of freezing and thawing and no important influence on either the compressive or flexural strength. It was concluded that five per cent would make a suitable specification limit on shale, provided that the total amount of shale and similar substances does not exceed this amount.

Apparently very little has been published on the effects of shale on concrete durability since the early 1930's. Thus present-day specifications for shale in concrete aggregate are based on the fragmentary information provided by the aforementioned studies plus actual service experience in the various states. The consensus of the results of the studies mentioned above seems to be that shale in small percentages (perhaps as much as ten per cent) doesn't cause severe damage to concrete exposed to freezing and thawing, but that surface pitting and popouts do result from the shale. These studies showed that some shales act differently than others but did not

suggest any methods for differentiating "good" shales from "bad" shales.

As early as the 1930's many state highway departments had specifications limiting the percentage of shale allowable in concrete aggregate. S. Walker and Proudley (67) noted 22 states which limited either shale or "soft, friable fragments and other deleterious particles including shale." Twelve of the states had definite specifications on shale itself in coarse aggregate with most of these allowing from 0.5- to 1.0-per cent shale. Nineteen of the states had specifications on soft, friable fragments including shale with most of these allowing about 5.0 per cent. These specifications were based on experience and on the early research outlined above.

Today an even greater number of states have specifications governing the percentage of shale in concrete aggregates. A recent survey (53) of the specifications of 46 state highway departments showed 23 with limits on the allowable percentage of shale; 29 with limits on the percentage of "soft fragments" (including shale). Those specifications which imposed no numerical limits generally contained a statement such as: "Coarse aggregate must consist of hard, strong, durable pieces." For those states which had a definite requirement for shale, the percentages allowed varied from 0.5 to 2.0 per cent with most states allowing about 1.0 per cent.

Although most of these specifications have been in effect for quite a few years and are based to a certain extent on experience and on the small amount of research that has been done on shales, there is some doubt as to how realistic these specifications are. For

example, none of the specifications takes into account differences in types of shales within the area governed by the specifications. Also, the maximum percentages of shale generally allowed by these specifications are considerably lower than the percentages found not harmful in the aforementioned studies by the Kentucky and Tennessee Highway Departments.

STATEMENT OF PURPOSE

The three major purposes of this study may be listed as follows:

1. The first objective was to learn more about those properties of the deleterious constituents of Indiana's aggregates which relate to the durability of concrete in which these deleterious materials make up part of the aggregate. Although these materials have received considerable study in the past, there is still much to be learned about their basic properties. For example, little petrographic work has been done on Indiana's chert and shale aggregates to determine more about their mineralogy, texture, and microstructure. Also, much has yet to be learned about the pore structures of cherts and shales.

2. In the past little has been done to differentiate between deleterious materials that are of the same general type, but are obtained from different geographic areas. A purpose of this study was to determine if the basic properties of cherts and shales from one part of the state differ significantly from those of cherts and shales from other parts of the state, and if there are significant differences in the properties of these materials, to attempt to determine if these differences also result in differences in durability of the materials.

3. A third objective of this study was to determine whether the present specifications of the State Highway Department of Indiana on

deleterious substances categorize aggregates on a realistic basis. Although these specifications are based on years of experience and research, they do not take into account possible differences in the durabilities of materials of the same type but from different areas.

SCOPE

This investigation was conducted primarily as a study of the durability of concrete produced by combining small percentages of deleterious materials from Indiana gravels with a single standard fine aggregate, crushed stone coarse aggregate, and portland cement. The fine and coarse aggregates both had good service records. In addition to the durability testing, tests were conducted to determine many of the basic physical properties of the deleterious materials in order to develop possible relationships between these properties and the durabilities of the materials. Cherts and shales were selected as the deleterious substances to be studied since these rock types are probably the most abundant of the deleterious constituents in Indiana's gravel aggregates, and because there is still much to be learned about the physical properties of these two materials and about the effects of these materials on the durability of concrete.

The cherts and shales were obtained from several gravel sources throughout the State of Indiana. The cherts and shales were the only components of the gravels that were studied. Although complete mineralogic analyses were conducted on the gravels from all the sources, this information was used only to give a general measure of the durability of aggregates from different areas within the state. Except in a general way, no attempt was made to determine from which geologic formations the gravel aggregates originated.

Tests of Basic Properties of Deleterious Materials

The cherts and shales were separated from their source gravels by hand-picking, and then were divided into specific gravity groups by means of heavy-liquid separation. Small random samples from these groups were then subjected to various basic tests. Bulk specific gravity, bulk specific gravity (saturated surface-dry basis), and apparent specific gravity were determined using a variation of ASTM Designation: C127-42, Method of Test for Specific Gravity and Absorption of Coarse Aggregate (1). True specific gravity was measured by pycnometer tests, and porosity was calculated using the bulk and true specific gravities. For the chert groups, a measure of the per cent of voids greater than 5 microns in diameter was obtained by microscopic study of polished thin sections utilizing a variation of the linear traverse technique of void study reported by Fears (12). The voids with diameters of 5 microns or less[1] are thought to be the critical ones in freeze-thaw deterioration of cherts (59). The volume of these very small voids was determined by subtracting the volume of voids greater than 5 microns from the total voids volume. Vacuum absorption, 24-hour absorption at atmospheric pressure, and rate of absorption at atmospheric pressure were determined on the same samples that were subjected to the specific gravity tests.

Petrographic studies of thin sections of the cherts and shales were also carried out. These studies were undertaken to determine such basic properties as grain size, microstructure, orientation of grains,

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[1] Voids 5 microns or less in diameter will be referred to as "Microvoids" in this report.

and mineralogy. No quantitative microscopic mineralogic analyses were undertaken, and no attempt was made to determine the origins of the cherts or shales.

Tests Performed on Concrete Beams Containing Cherts or Shales

Testing of concrete containing small percentages of chert or shale samples consisted primarily of freeze-thaw testing. Blends of two, four, ten, and, in some cases, six per cent of each type of chert and shale were made with a standard crushed limestone, and these blends were used to make 3- by 4- by 16-inch air-entrained concrete beams. These beams were then subjected to up to 300 cycles of freezing and thawing in an automatic freeze-thaw machine.

Average ASTM durability factors at 300 cycles of freezing and thawing were computed for the various combinations that were tested. Where possible, statistical analyses were conducted on the durability factors to determine the significance of the effects of variables such as the sources of the deleterious materials and the per cents of these materials used. In addition, tests were conducted to determine the effects of bubble spacing factors and specific surface areas of the air voids on the freeze-thaw durability of air-entrained concrete.

Since the beams containing shale showed little deep-seated deterioration when exposed to 300 cycles of freezing and thawing, studies of surface deterioration of these beams were carried out. These studies consisted of visual evaluation of "popouts" and surface pitting due to freeze-that deterioration of the shale. A similar study was also undertaken for the beams containing chert.

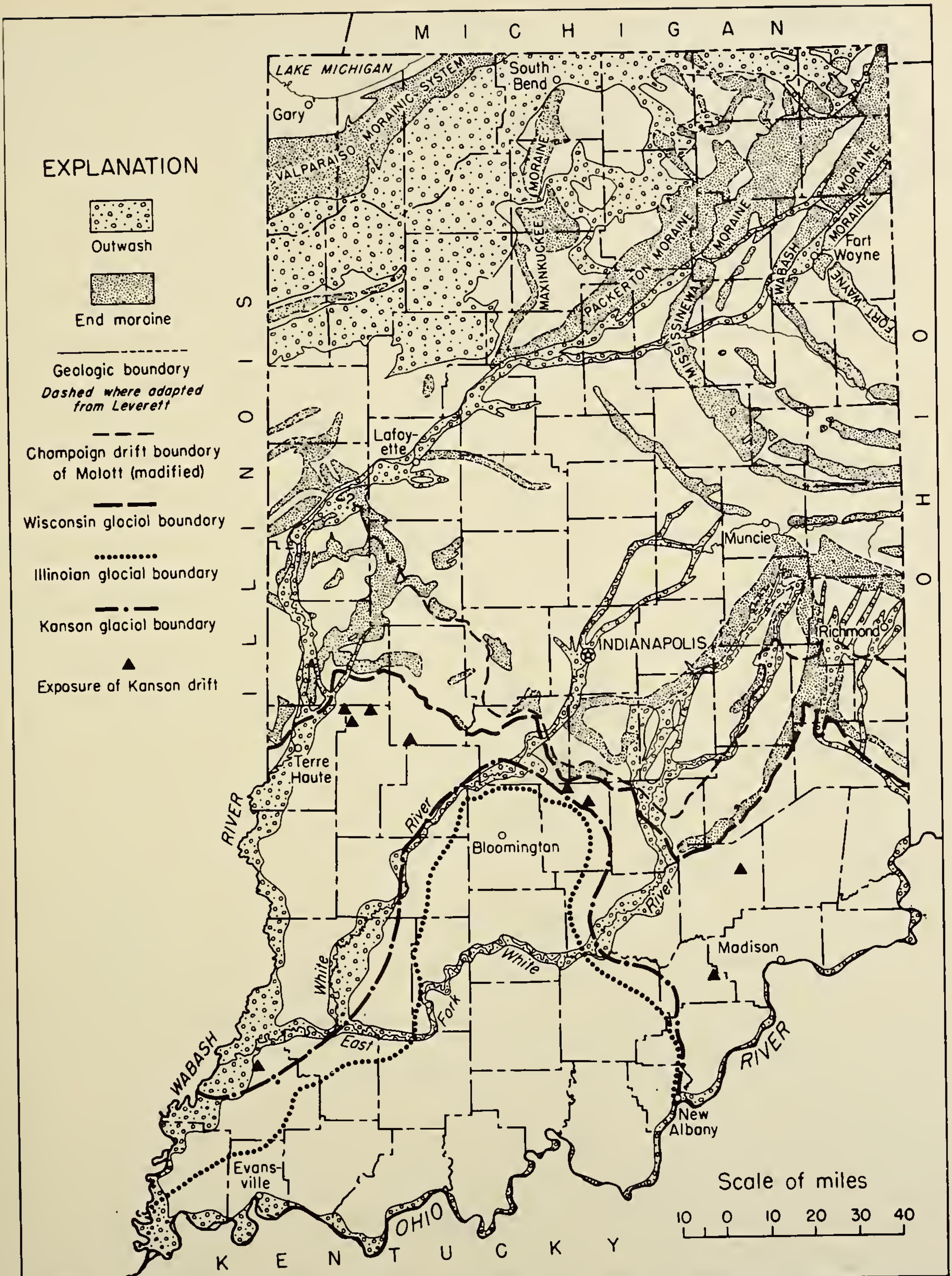
CHERTS AND SHALES IN INDIANA GRAVELS

The gravel deposits of Indiana are almost entirely of glacial origin, having been eroded from bedrock by continental glaciers of Kansan, Illinoian, and Wisconsin ages, and deposited by glacial meltwater or reworked and deposited by later streams. Most of these glacial gravels occur either as outwash sediments, i.e., outwash-plain deposits and valley-train deposits, or as ice-contact sediments, which include moraine, kame, and esker deposits. Of these, the valley train deposits provide the greatest percentage of Indiana's commercial gravel resources. The distribution of glacial deposits in Indiana is shown in Figure 1.

Distribution of Cherts and Shales in Indiana Gravels

The composition of a gravel deposit depends on the types of bedrock which have been eroded by the glacier supplying the gravel. In places where the ice moved over brittle shales or sandstones, material from these rocks is present in glacial deposits near the shale-sandstone source and can be found for a few miles in the direction of ice movement (32). Shale particles are not ordinarily found far from their bedrock source because they are soft and extremely susceptible to abrasion.

Where glaciers moved over limestone or dolomite bedrock, a large amount of these materials is found in the direction of ice movement. If the limestone is cherty, considerable chert may be found in the gravels. Unlike shale, chert may be carried farther from its source area because



AFTER WAYNE (69)

FIGURE 1. MAP OF INDIANA SHOWING GLACIAL BOUNDARIES AND WISCONSIN GLACIAL DEPOSITS

it is extremely resistant to abrasion. Figure 2 shows the dominant lithologies of bedrock in Indiana and presents the general location of bedrock sources of some of the rock types in Indiana gravels.

Figure 3 illustrates the distribution of cherts and shales throughout Indiana. This figure is based on mineralogic analyses of Indiana's gravels by the Indiana Geological Survey (32) and by the author. Comparison of this figure with Figures 1 and 2 shows that the highest concentration of shale gravels occurs along the late Wisconsin (Cary) Valparaiso Moraine. These shale gravels have been derived largely from the area of Devonian and Lower Mississippian shales in the extreme northern part of the state as shown in Figure 2. Smaller percentages of shales that have been derived from local shale bedrock are found in gravel deposits in several areas throughout the state.

Gravels containing chert are distributed throughout most of Indiana. Since cherts are very resistant to abrasion, the chert distribution does not show as much bedrock control as that of the shales. Worthy of note are the heavy concentrations of chert in the gravels of the Ohio and lower Wabash Rivers and the very low incidence of chert gravels in the southeastern part of Indiana. The concentrations of this material along the Ohio and lower Wabash Rivers are a result of large amounts of chert occurring as nodules and lenses in carbonate bedrock units in areas drained by these streams.

It also should be noted that percentages of cherts or shales will vary somewhat from one part of a gravel pit to another. Along the Ohio River it has been found that the chert content of river gravels will vary considerably from one gravel bar to another and will even show

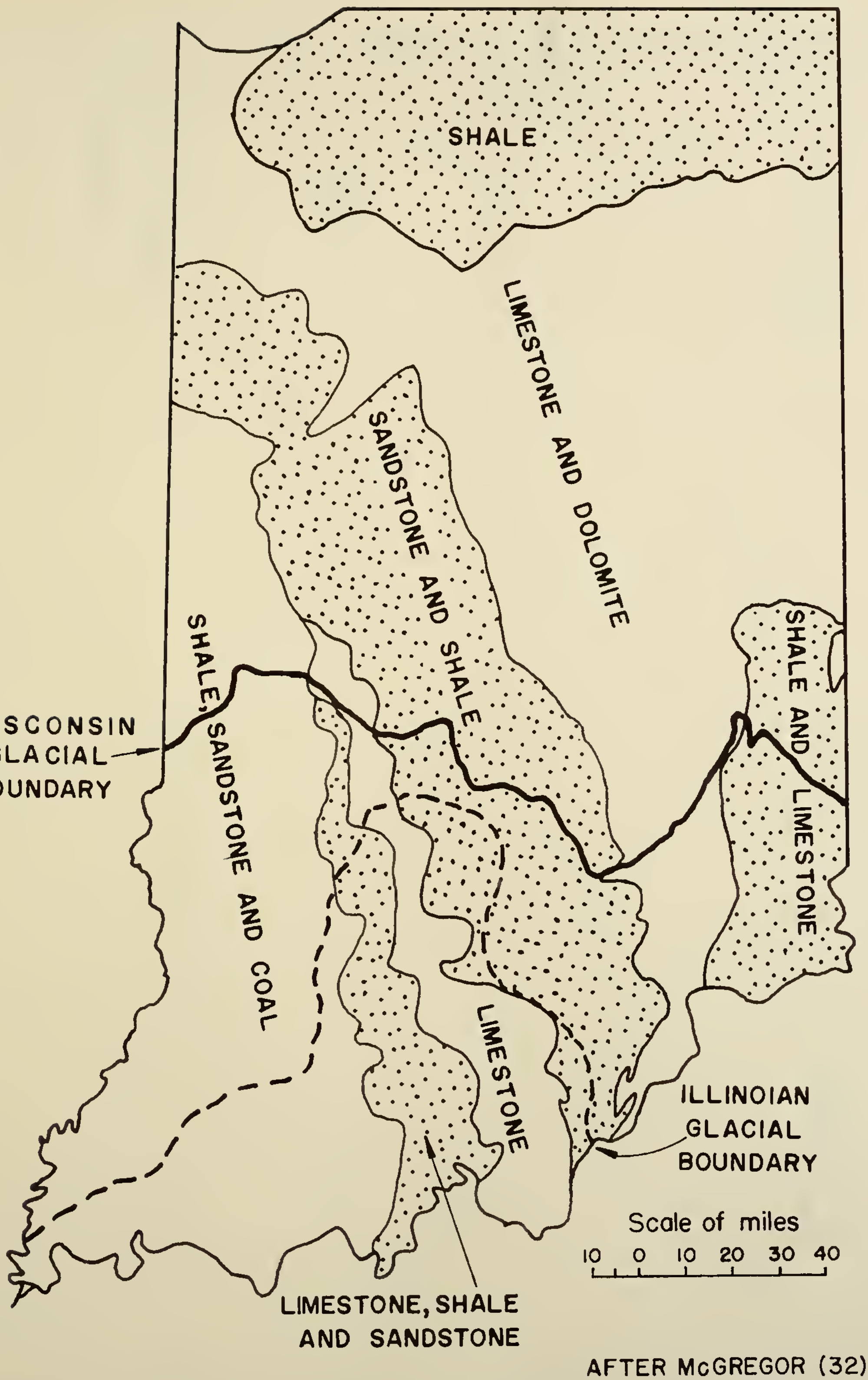


FIGURE 2. MAP OF INDIANA SHOWING DOMINANT BEDROCK LITHOLOGIES

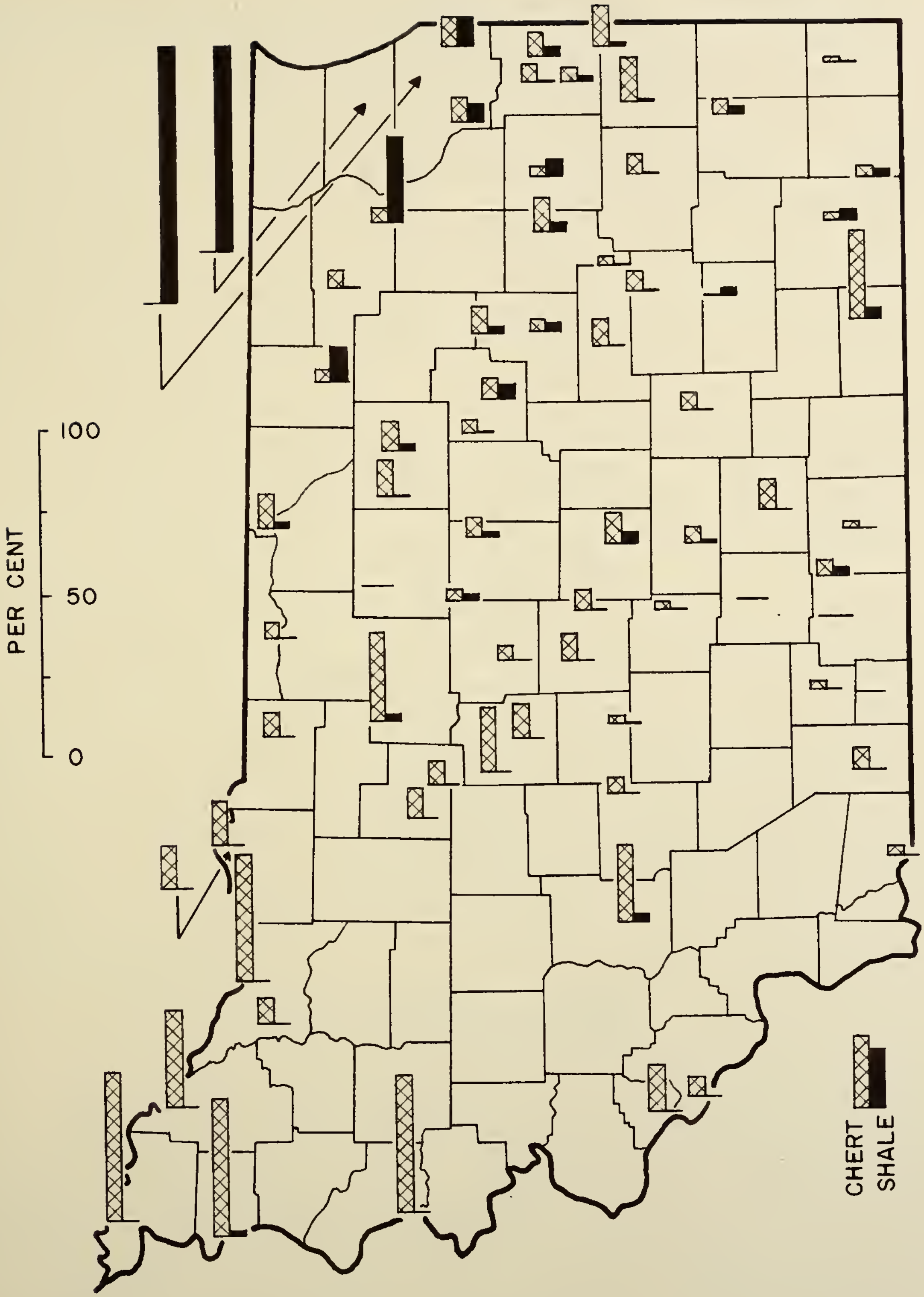


FIGURE 3. MAP OF INDIANA SHOWING APPROXIMATE PERCENTAGES OF CHERT AND SHALE IN GRAVEL DEPOSITS

considerable variability from one part of the same bar to another.

Selection of Sources of Cherts and Shales

Only gravels containing sufficiently large percentages of chert or shale were selected for this study to permit the needed quantities of these materials to be handpicked from the gravel in a reasonable amount of time. This eliminated many gravel deposits as possible sources of either chert of shale.

Also, it was planned that the sources selected be widely distributed throughout Indiana so that they would originate from different bedrock units and would represent as widely divergent a group of cherts and shales as can be obtained within the state.

The chert and shale sources were selected on the basis of these criteria and the information presented in Figures 1, 2, and 3. These sources are listed in Tables 1 and 2, and their geographic locations are shown in Figure 4. Mineralogic analyses of the gravels from these sources plus analyses for a few other deposits in Indiana are presented in Table 22 in Appendix A.

Sampling the Cherts and Shales

The samples of chert and shale to be used in this study were obtained by handpicking the materials from commercial stockpiles of aggregates. Pieces ranging in size from 3/8 inch to 1 inch were selected. Every attempt was made to obtain completely random selection within this size range. After the samples were brought in from the field, they were carefully checked in the laboratory to ascertain that all material used was chert or shale.

Table 1

Locations and Geologic Sources of Chert Gravels

Sample Number	Location	Geologic Source
2063	Decatur	Valley train of St. Mary's River
2064	Elkhart	Valley train of St. Joseph River
2066	Seymour	Valley train of East Fork of White River
2067	West Lafayette	Valley train of Wabash River
2072	Gosport	Valley train of West Fork of White River
2077	Ohio River near Tell City	Gravel bar in Ohio River

Table 2

Locations, Geologic Sources, and

Descriptions of Shale Gravels

Sample Number	Location	Geologic Source	Description
2063	Decatur	Valley train of St. Mary's River	Hard carbonaceous silty shale
2066	Seymour	Valley train of East Fork of White River	Soft, platy weak shale
2068	Near LaPorte	Kame on Valparaiso Moraine	Very soft argillaceous shale
2075	Indianapolis	Valley train of White River	Fairly hard carbonaceous shale
2076	South Bend	Valley train of St. Joseph River	Weak, platy carbonaceous shale

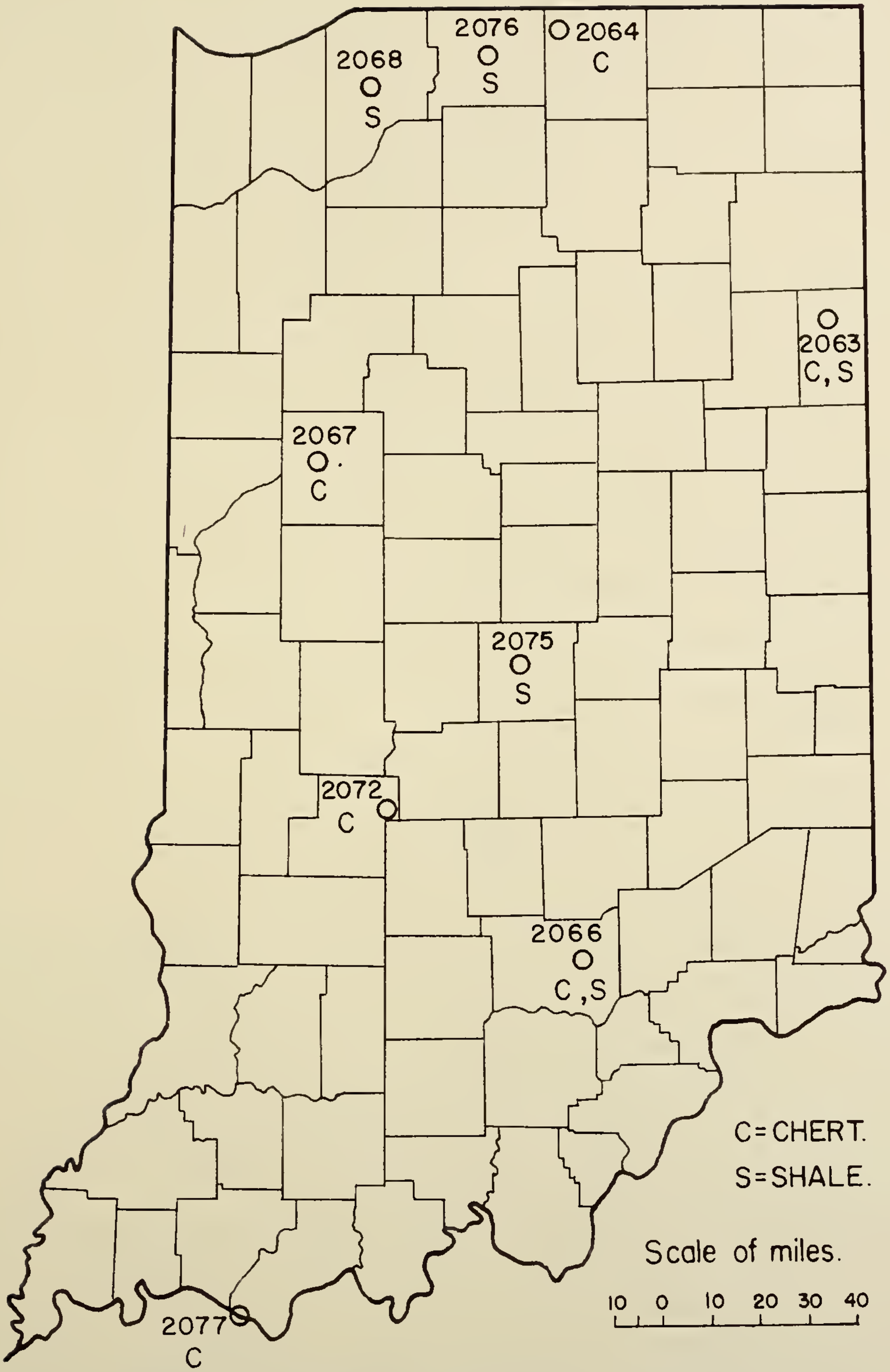


FIGURE 4. MAP OF INDIANA LOCATING SOURCES OF CHERT AND SHALE GRAVELS USED IN THIS STUDY

DESCRIPTION OF TESTS OF BASIC PROPERTIES OF CHERTS AND SHALES

It has become increasingly apparent in recent years that aggregates are chemically and physically active in ways that exert a critical control on their serviceability when used in concrete. For this reason it has become imperative that in any study of the durability of concrete aggregates as much information as possible be gained on the basic properties of the material in question. The tests of the basic properties of cherts and shales used in this study may be divided into the following groups: (a) specific gravity tests, (b) porosity determinations, (c) absorption tests, and (d) petrographic studies.

Specific Gravity Tests

The various specific gravity tests are of use in a study of the durability of aggregates primarily because the results of these tests provide information on the pore characteristics of the aggregates. The types of specific gravities determined in this study were: (a) bulk specific gravity (saturated surface-dry basis) determined by heavy-liquid separation, (b) bulk specific gravity, and (c) true specific gravity.

Fractionation by Heavy Liquids

Heavy-liquid separation was performed on each of the six cherts

listed in Table 1 and the five shales listed in Table 2. After being separated into 3/8 to 1/2-inch, 1/2 to 3/4-inch, and 3/4 to 1-inch size ranges, the cherts were divided into specific gravity groups (bulk saturated surface-dry basis) within these size ranges by means of heavy liquid separation. The liquids used were carbon tetrachloride, specific gravity 1.58, and acetylene tetrabromide, specific gravity 2.97. For the chert separation, these compounds were mixed together to give liquids with specific gravities of 2.35, 2.45, and 2.55. The chert samples were immersed in water for 24 hours, then placed in the 2.55-specific gravity liquid. The portion that floated was skimmed off, and the procedure was repeated on this portion in the 2.45- and 2.35-specific gravity liquids. In this way the cherts were separated into the following specific gravity ranges: 2.55 plus, 2.45-2.55, 2.35-2.45, and 2.35 minus. The oven-dry weight of each specific gravity group was recorded and the per cent by weight within each of the three specific gravity groups was calculated. After 1000-gram samples of each chert were tested in this way to determine the specific gravity distribution of the chert from each source, it was decided to separate the entire chert supply into specific gravity groups and to run all succeeding tests on material from each group. Since there was little material in the 2.35 minus fraction, it was decided to combine the 2.35-2.45 and 2.35 minus groups. As a result, all the chert was separated into 2.55 plus, 2.45 - 2.55, and 2.45 minus specific gravity groups,(bulk saturated surface-dry basis) for use in further tests.

Specific gravity distributions (bulk saturated surface-dry basis) were also obtained for the shales by the heavy-liquid separation

technique. A sample weighing approximately 1000 grams was obtained from each of the shales by quartering, and the specific gravity distribution was determined for each of these samples. The shale samples were separated into the following specific gravity ranges: 2.55 plus, 2.45-2.55, 2.35-2.45, 2.25-2.35, 2.15-2.25, 2.05-2.15, and 2.05 minus.

On the basis of the specific gravity distribution obtained from the shales, a mean specific gravity value was calculated for each shale to be tested. Then by separating at two specific gravities, one slightly below and one slightly above this mean value, it was possible to obtain a small sample of each shale having a specific gravity very close to that of the mean for the whole supply of that particular shale. Several tests of basic physical and chemical properties were later run on these small shale samples with narrow specific gravity ranges representing the average bulk specific gravity (saturated surface-dry basis) of the shale in each source.

Determination of Bulk Specific Gravity

Bulk specific gravity tests were conducted on five samples of shale and six of chert. Although all of these samples had previously been separated into bulk specific gravity groups (saturated surface-dry basis) by heavy-liquid separation, additional specific gravity tests were run in order to determine accurately the bulk specific gravities of the samples for use in calculating the total porosities of the aggregates. These tests were run on 1/2 to 3/4-inch pieces which represented the median size range used in this investigation.

The five shale samples tested were those having the mean bulk

specific gravity (saturated surface-dry basis) obtained from each of the sources listed in Table 2. In the case of chert, material from two of the original six cherts (sources 2067 and 2077) listed in Table 1 was further tested for basic physical properties. Since these cherts had been separated into three specific gravity groups, six samples were tested for bulk specific gravity: one for each of the three specific gravity groups from sources 2067 and 2077.

To determine the bulk specific gravities of these samples, a technique similar to that suggested by Metcalf (33) was used. Samples weighing about 200 grams were immersed in water for 24 hours at about 70°F. At the end of that time they were weighed in water at 70°F. while suspended from an analytical balance. The basket in which the samples were immersed was also weighed in water before the tests began.

After weighing in water, the samples were carefully blotted to a surface-dry condition, and the saturated surface-dry material was weighed in air. This operation was performed as rapidly as possible to prevent evaporation of water from the pores.

The bulk specific gravity was calculated from the following equation:

$$S_b = \frac{W_o}{W_{ssd} - W_1 + W_2}$$

where

W_o = oven-dry weight of aggregate,

W_{ssd} = saturated surface-dry weight of aggregate,

W_1 = submerged weight of aggregate and basket,

W_2 = submerged weight of basket.

The bulk specific gravity on saturated surface-dry basis was

determined as follows:

$$S_{bssd} = \frac{W_{ssd}}{W_{ssd} - W_1 + W_2}$$

where the abbreviations are the same as those used in finding the bulk specific gravity above.

Determination of True Specific Gravity

The true specific gravities of the chert and shale samples were determined by means of a slight modification of the pycnometer method used in specific gravity testing of soils as outlined by Lambe (22). For this test a random sample of about ten grams of powdered material was obtained from each of the six chert and five shale samples tested for bulk specific gravity. This material was powdered by first crushing it in a small jaw crusher, and then reducing it to a powder by means of a diamond mortar. All the material tested passed the #100 sieve.

Because of the small quantities of material involved, a 50-ml. pycnometer was used in place of the 500-ml. volumetric flask commonly specified for soil studies. The 50-ml. pycnometer was calibrated using distilled water at various temperatures. A calibration curve relating the weight of the pycnometer plus water to a range of temperatures from 20° to 30°C. was plotted. All weighings were done on an analytical balance to the nearest 0.001 grams.

After oven-drying to constant weight, each of the powdered samples was cooled in a desiccator, and weighed rapidly to prevent absorption of water from the atmosphere. Each sample was then placed in the pycnometer with care being taken that no material was lost in transfer. Sufficient distilled water to completely cover the pulverized chert or shale was then introduced into the pycnometer which was rolled on a flat surface in a tilted position to eliminate as many air bubbles as possible. The pycnometer was then evacuated by means of a water-jet aspirator for several minutes to remove any air left after the rolling procedure. Following evacuation, the pycnometer was filled nearly to the top with distilled water, which previously had been boiled to remove all air, and allowed to set for about 30 minutes so most of the powdered material would settle somewhat and little would be lost when the stopper was inserted and a small amount of water overflowed. The stopper was then inserted with care being taken that no air bubbles were entrapped. The outside of the pycnometer was dried carefully and the pycnometer plus water and aggregate were weighed. At the same time the temperature of the water and pulverized aggregate was read by means of a thermometer in the pycnometer stopper. Since the distilled water was kept at room temperature before the tests, and since the tests were run at room temperature, the insertion of the thermometer (which was also at room temperature) had little effect on the temperature of the water and powdered aggregate which could be accurately read to 0.1°C.

After weighing the pycnometer and its water-aggregate mixture, the powdered aggregate was removed from the pycnometer, placed in an evaporating dish, and dried to constant weight under heat lamps and in an electric

oven. After cooling in the desiccator, these samples were weighed to provide a check on the original oven-dry weight.

True specific gravity was computed from the following equation:

$$S_s = \frac{S_w W_o}{W_o - W_1 + W_2}$$

in which

S_s = true specific gravity of aggregate (specific gravity of solids),

S_w = specific gravity of water at temperature of test,

W_o = weight of oven-dry powdered aggregate,

W_1 = weight of pycnometer containing powdered aggregate and water,

W_2 = weight of pycnometer filled with water (taken from the calibration curve for pycnometer).

Porosity Determinations

The lack of freeze-thaw durability of an aggregate in concrete is primarily dependent upon the ability of the aggregate to become and stay saturated while undergoing freezing and thawing (27). The pore characteristics of an aggregate determine the amount of water an aggregate can absorb, the rate of absorption, and ease of drainage. Apparently the size of the aggregate voids is critical in freeze-thaw deterioration. Rhoades and Mielenz (46) stated in this connection:

> ..."Water moving by capillarity will not enter aggregates containing only large voids, even if these voids are interconnected and penetrable. On the other hand, small voids will be penetrated; and if these openings are smaller than those of the cement paste, the water will be preferentially drawn into them from the paste. During periods of hydration or drying, while water is being withdrawn from the interstices of cement paste, water will be drawn by capillarity

> from aggregate particles containing only voids larger than those in the paste, but the last residuals of water remaining in relatively dry concrete may be concentrated in aggregate-voids smaller than those in the cement paste. Thus rocks containing exceedingly small, interconnected voids are capable of attaining and retaining a high degree of saturation in concrete, and may be susceptible to disruption if repeated freezing occurs."

Blanks (5) found that, under natural conditions of freezing, voids less than 5 microns in diameter, and particularly less than 4 microns in diameter, will drain effectively only at hydrostatic pressures that exceed the tensile strengths of some rocks and concrete. Sweet (59) used 5 microns as the critical pore diameter, and found that for unseparated Indiana aggregates, the volume of voids less than 5 microns in diameter for Indiana limestones with good service records was less than 0.057 (expressed as a ratio of void volume to bulk volume), and it was greater than 0.091 for those with poor records. Fears (13) also reported correlation between the volume of pores less than 5 microns in diameter and the freezing-and-thawing durability of Indiana limestones.

Since porosity is so important in the freezing-and-thawing durability of concrete aggregates, it was deemed necessary to carry out several studies of voids in the cherts and shales used in this investigation. Therefore total volume of voids and volume of voids less than 5 microns in diameter were determined for the six chert and five shale samples being studied.

Total Porosity

The total porosity, or ratio of voids to bulk volume of a sample

of aggregate, was calculated from the following relationship between bulk specific gravity and true specific gravity (derivation of which may be found in Appendix C):

$$n = \frac{V_v}{V} = 1 - \frac{S_b}{S_t}$$

where

n = porosity,

V = total volume,

V_v = volume of voids,

S_t = true specific gravity,

S_b = bulk specific gravity.

Size-Studies of Pores

The per cent of total volume made up of voids less than 5 microns in diameter (microvoids) was obtained by subtracting the per cent of bulk volume of the sample comprised of voids greater than 5 microns in diameter from the total porosity of the sample. The volume of voids greater than 5 microns in diameter was determined by microscopic analysis of the voids combining the techniques of Verbeck (64), who used a camera lucida method for measuring air voids in hardened concrete and Rosiwal (48), who developed a linear mensuration method for determining the percentages of different types of minerals in thin sections of rocks.

The actual method employed in this study involved the use of polished sections and application of linear traverse techniques. Other studies (13) had demonstrated that study of pores in thin sections between crossed nicols does not delineate pores less than

about 30 microns in diameter. In this case, a Hunt-Wentworth recording micrometer of the type commonly used for micrometic mineralogical analyses was used to make the linear measaurement. A magnification of 100x was required to define pores 5 microns in diameter. A similar use of the linear traverse technique is reported by Fears (12) wherein he studied air-voids in hardened concrete.

Since shales will not take a satisfactory polish, the study of polished sections was limited to six chert samples, one for each of the three chert specific gravity groups for sources 2067 and 2077. The study was performed on polished surfaces of these pebbles selected at random from the 1/2 to 3/4-inch grading in each of the specific gravity groups. The procedure for study of the polished sections was as follows:

1. A section having two plane and approximately parallel surfaces was cut from each piece of chert using a diamond rock saw. These surfaces were smoothed off using steel lapping wheels and three grades of silicon carbide grinding medium (Nos. 120, 320, and 600). Final polishing was done by hand on a hard plane glass plate 12 inches by 12 inches using centriforce abrasive optical polishing powder. Water was used as a lubricating and dispersing medium during grinding and for washing the polished sections.

2. The voids intersected by the plane of the polished surfaces were filled with red pigment by rubbing the surfaces with powdered cuprous oxide and wiping off the excess. The voids thus appeared red when the surface was viewed with the microscope.

3. The polished surfaces were viewed under a petrographic microscope in reflected light at 100x magnification. The Hunt-Wentworth micrometer has a traversing mechanism driven by six calibrated wheels each of which may be used separately to record movement across different elements in the traverse. In this study, parallel traverses at equal intervals were made across the polished surfaces of the pieces of chert and shale. One wheel was used to record the length of traverse in which voids were intersected; one or two other wheels were used to record the length of the traverse path in solid areas. At the end of each traverse, the dial readings for length of traverse in voids and length in solids were recorded. In keeping with the purpose of this study, only the intersected voids larger than 5 microns in diameter were included as part of the void measurement. Separation of the voids at the 5-micron level was accomplished by means of a micrometer eyepiece. In running the traverse, the end of one of the markings on the micrometer scale was used to denote the path of traverse.

4. After the required number of traverses were completed for each surface, the percentage of voids was calculated by dividing the total traverse path in voids by the total traverse path in voids plus the total traverse path in solids.

Ten equally spaced traverses were run on each of three polished surfaces from each sample. Since this is fewer than the 15 traverses Heinrich (16) states are required on the average thin section in order to obtain an accuracy of about one per cent in mineral analyses

of granular rocks, and since only three pieces were studied from each sample, the average percentages of voids determined in this study should be considered only as approximations of the true percentages.

Once the porosity consisting of voids greater than 5 microns in diameter was determined by this technique, the portion of total porosity consisting of voids less than 5 microns in diameter (micro-voids) a size thought to be critical in determining the quality of concrete aggregate, was calculated by subtracting the porosity for the voids greater than 5 microns in diameter from the total porosity as determined from the bulk and true specific gravities.

Absorption Tests

Although the porosity of an aggregate can be an indication of its durability, more basically the freeze-thaw durability of an aggregate is thought to be more closely related to the ability of these pores to absorb and retain water while the particle is undergoing freezing and thawing. Absorption and fluid-flow measurements therefore are fundamental to this type of study (11).

The absorption tests carried out on cherts and shales in this study were (a) vacuum-saturated absorption, (b) 24-hour absorption at atmospheric pressure, and (c) rate of absorption at atmospheric pressure for a period of one week.

Vacuum-Saturated Absorption

Studies by Sweet (59) have shown that 24-hour immersion at atmospheric pressure does not approximate the high degree of saturation that many river gravels have at the time of their production for aggregate. To obtain absorption values similar to the worst conditions of field saturation, it appears necessary to evacuate the dry aggregate and immerse it in water while in this condition.

A vacuum system consisting of a sealed chamber connected by individual valves to (a) a manometer, (b) a container of water free of air bubbles, and (c) a vacuum pump of the water-jet aspirator type was used. Dry aggregate was placed in the chamber and evacuated at a pressure of approximately 2.3 cm. of mercury for a period of one hour. At the end of this period, water free of air bubbles was admitted to the chamber with the vacuum still maintained. The aggregate was covered by water and left in an immersed condition for 23 hours. Using this same type of equipment, Sweet (59) obtained a degree of saturation of approximately 97 per cent for crushed limestone aggregate.

At the end of the 24-hour period, the samples were removed from the vacuum saturator, blotted to a surface-dry condition and immediately weighed to the nearest 0.01 gm. They were then dried in an oven at 105°C. for 24 hours, allowed to cool in a desiccator, and again weighed. The vacuum absorption expressed as a percentage was calculated as follows:

$$\text{Per cent vacuum absorption} = \frac{W_s - W_o}{W_o} \times 100$$

where

W_s = saturated surface-dry weight of sample, in grams,

W_o = oven-dry weight of aggregate in grams.

Vacuum-saturated absorption studies were conducted on 54 chert samples and 15 shale samples. Each chert sample was taken from one of the three gravity groups from each of the three size ranges of the six chert sources. Each shale sample was taken from one of the three size ranges of the five shale sources.

In addition to the absorption studies previously discussed, vacuum-saturated absorption tests were also conducted on a few pieces of broken chert pebbles taken from beams with very low durability factors after they had completed the freeze-thaw test. Each of the pieces of chert tested was intersected by a deep-seated crack in its particular beam and was thought to be the cause of the crack. These individual pieces were studied to determine if their absorptions were any greater than those of the average pieces from their particular groups. The pieces studied were all from the 2.45 minus bulk specific gravity groups (saturated surface-dry basis) because this "light-weight" chert was the only type resulting in deep-seated cracks. Only one pebble was tested from each chert source, with the exception of source 2077 where it was thought that a few extremely porous pieces might be causing most of the failures. Three pieces from this source were tested.

Rate of Absorption at Atmospheric Pressure

Studies such as absorptivity and tortuosity of the pore system,

which are related to permeability, often are undertaken as an integral part of studies of the pore characteristics of aggregates. Such studies, however, are not easily performed on gravels because of their irregular shapes. It has been shown that similar information can be obtained from the simple rate of absorption test which is extremely useful in comparing the relative permeabilities of different aggregates (11).

The test consists simply of immersing the aggregate in water at atmospheric pressure, removing the aggregate from the water at predetermined intervals, and rapidly blotting it to a surface-dry condition following which it is weighed and immediately returned to the water. In this test, oven-dry weights were determined before immersion and at completion of the test. The intervals selected were 5, 10, 20, 40, 80, etc., minutes to a maximum of 5,120 minutes. The total elapsed time was seven days. The method of calculation was the same as that used for the simple 24-hour absorption tests with one exception. Some of the shale samples, notably 2068, were so soft that they lost a little material each time they were blotted to a saturated surface-dry condition. This slight loss was taken into account in the calculations by assuming an equal loss each time the blotting operation was carried out. Since the original oven-dry weight and the final oven-dry weight of each sample were determined, it was a simple matter to determine a theoretical oven-dry weight for the end of each interval on the basis of this assumption.

After determining all the absorption values, a plot of percentage of absorption versus elapsed time was made on semi-logarithmic graph

paper for each sample.

Petrographic Studies

Microscopic petrography has long been a valuable tool in the study of the characteristics of rocks. Less than 30 years ago, however, petrography began to find a place in the study of concrete agggregates. Runner (49), in 1937, was one of the first to apply petrography to the study of deleterious substances in aggregates. His petrographic investigations were followed by reports of similar studies and descriptions of techniques by Mielenz (34, 35) Rhoades and Mielenz (46, 47), and Mather and Mather (31). However, none of these studies attempted to find a relationship between the results of petrographic studies of deleterious materials and the laboratory freeze-thaw durability of these materials; this is one of the primary purposes of the present investigation.

The petrographic study was carried out using a Leitz Ortholux petrographic microscope with binocular attachments. This microscope may be used for study with either transmitted or reflected light at magnifications ranging from 25x to 750x.

Thin sections were made from three pieces from each of the five shale samples and six chert samples subjected to the basic tests of physical properties previously described. The thin sections were studied under transmitted light at magnifications of approximately 100x to 400x. The properties studied were (a) mineralogy, and (b) textures and microstructures.

Mineralogy

Chert is composed primarily of silica which characteristically takes the form of (a) feathery chalcedony, (b) cryptocrystalline quartz, (c) microcrystalline quartz, (d) opal, and (e) cristobalite (16). Older cherts or somewhat metamorphosed cherts contain little or no chalcedony or opal, and are characterized by microcrystalline quartz. Many cherts are relatively impure, containing abundant calcite, with occasional rhombs of dolomite or siderite. Such rocks, poorly termed porcellanites, may grade into cherty limestone.

The petrographic study of cherts was intended to provide qualitative information as to the types of silica minerals and impurities present in Indiana cherts and the relative abundance of these types. Any possible relationships between mineralogy and durability of the aggregates were investigated.

The complete mineral composition of shales, as of clays, is not easily determined microscopically. In fact, most of what is now known about shale mineralogy is based upon chemical, thermal, x-ray, and electron microscope studies (71). The coarser shale particles, which may consist of quartz, orthoclase and plagioclase, muscovite, chlorite, and accessory hornblende, biotite, epidote, magnetite, tourmaline, and zircon, are set in a "paste," a microcrystalline to cryptocrystalline matrix of clay minerals, quartz, sericite, chlorite, limonite, rutile needles, and carbonaceous material (16). Other possible constituents are pyrite, glauconite, collophane, and carbonate grains. The clay minerals include illite (probably the

most common), chlorite, montmorillonite, and kaolinite.

The main mineralogical varieties of shale are (a) quartzose shale with calcareous, ferruginous, carbonaceous, or even glauconitic material in the matrix; (b) feldspathic shale, containing greater than ten per cent silty feldspar and with considerable kaolinite in the matrix; (c) micaceous shale with abundant detrital muscovite and considerable matrix sericite; and (d) chloritic shale with abundant silty feldspar and a chloritic matrix that may be highly carbonaceous (16).

Although the fine grain size of shales renders microscopic mineralogic analyses difficult, these studies were carried out along with texture and microstructure studies. Most of the effort went toward identification of silt-sized grains in the shales. In addition to microscopic mineral analysis, x-ray diffraction (7), and differential thermal analysis (20) were used to study the mineralogy of the shales.

Textures and Microstructures

The grain size and textural variability of cherts largely reflect the degree of crystallinity of the silica mineral. Cherts consist of mixtures in various proportions of mostly isotropic silica with scattered polarizing specks of chalcedony and cryptocrystalline quartz (16). Two types represent in a general way the limits of compositional and textural variations: (a) a type consisting of microcrystalline, equant, polyhedral, and uniformly sized quartz grains, and (b) a variety consisting of irregularly grained, fibrous

chalcedony. This study attempts to discover any relationship between chert textures and/or microstructures and the durability of the chert.

Shales do not lend themselves to microscopic grain size analysis because of the extremely small size of their grains (37). It is possible, however, to obtain qualitative information on shale textures by means of microscopic study of thin sections. Although clay-sized particles predominate in shales, most shales also contain a high proportion of silt-sized particles (16). In some shales the silt-sized particles may even predominate. There are a few very fine-grained shales, however, which have essentially no silt particles. The coarser clastic particles in a shale are generally subangular to subrounded.

Shales generally show a marked parallel arrangement of both the matrix constituents and the coarser detritals (16). Small-scale laminations, varves, or banding are common, resulting from deposition of varying amounts of such materials as carbonaceous shreds, quartz, calcite, or chlorite, or from deposition at different times of particles of varying grain sizes.

The individual grains in shales also tend to have a preferred orientation. Most of the grains lie with their "c" crystallographic axes oriented parallel to the bedding of the shale. Since the clay minerals in shales generally have the shape of thin, flat plates or rods, it is reasonable to expect a lesser porosity for a shale in which most of grains are oriented parallel to each other than for one in which the individual grains are oriented randomly. For clays, Lambe (23) has presented this relationship of porosity to orientation of particles, and

for newly deposited clays has related the amount of orientation to the origin of the clay, with salt water clays flocculating into a random arrangement with high porosity and fresh water clays dispersing in a parallel arrangement with low porosity. It is possible that this situation carries over to shale deposits, too, and that shale porosity may be related to mode of deposition, but it is more probable that compaction of the original clays during formation of shales results in orientation of grains of both fresh water and salt water deposits.

Regardless of the method by which the orientation of grains occurs, a study of this phenomenon in the five shale samples was initiated to learn more about the relationship between porosity and orientation of grains. A technique similar to that used by Mitchell (36) for optical study of the orientation of clay particles was used. Thin sections were cut parallel and perpendicular to the bedding of each shale, and these sections were studied between crossed nicols at 100X magnification. The optical properties of a randomly oriented group of extremely small particles between crossed nicols are indeterminate; such a group of particles has a uniform interference color when rotated under crossed nicols. If,however, the particles are aligned parallel to each other, they act as a unit and exhibit mass optical properties. For plate-shaped particles, the refractive indices in the direction of the "a" and "b" crystal axes are approximately equal, but differ considerably from the index of refraction along the "c" axis. If a thin section of parallel-oriented platy minerals is cut perpendicular to the "c" axes of the minerals, the refractive index

along the "c" axes is such that if viewed under polarized light, this section would appear uniformly gray while rotated through 360°. The refractive indices of "a" and "b" axes, however, are such that if thin sections are cut normal to these axes (parallel to "c" axes), four stages of illumination and extinction will be observed as the sample is rotated through 360°(36). Since platy clay minerals would tend to be oriented with their short "c" axes normal to the bedding of the shale, thin sections cut normal to the bedding were primarily used in this study. The results of the orientation study were compared to those of the porosity studies of shales.

EFFECT OF DELETERIOUS MATERIALS ON THE FREEZE-THAW DURABILITY OF CONCRETE

Experimental Design

In order to determine the effect of presence of the deleterious materials on freeze-thaw durability of concrete, small percentages of the cherts and shales being studied were incorporated in 3- by 4- by 16-inch air-entrained portland-cement concrete beams made with a standard portland cement, crushed stone coarse aggregate, and natural sand fine aggregate. The aggregates were obtained from sources of proven good quality. The only variables purposely introduced into the experiment were the deleterious materials themselves. Since the experiment design for cherts in the freeze-thaw study differed from that for shales, the two are presented separately.

Experiment Design for Chert Freeze-Thaw Study

An experimental outline was set up in which three variables were introduced into the production of beams containing chert with all other controlled factors remaining constant. The three variables were:

(a) <u>Source of Chert</u>. Material from each of the six sources of chert from throughout the State of Indiana was used.

(b) <u>Specific Gravity of Chert</u>. The chert from each of the six

sources was separated into three groups based on bulk specific gravity (saturated surface-dry basis). The specific gravity ranges selected for these groups were 2.55 plus, 2.45-2.55, and 2.45 minus. Separation was accomplished using mixtures of carbon tetrachloride (specific gravity 1.58) and acetylene tetrabromide (specific gravity 2.97). Beams were made containing chert from each source and at each level of specific gravity.

(c) Percentage of chert. Chert from each combination of sources and specific gravity groups was included with the crushed stone coarse aggregate in the beams in amounts of two, four, and ten per cent. During the course of the experiment, it became obvious that little deep-seated freeze-thaw deterioration of the beams would occur in any but those containing chert of the 2.45 minus range. In addition, it was found that even within this group, little deep-seated deterioration occurred in the two and four per cent blends, but there was considerable failure in the beams containing ten per cent of the lightweight chert. Therefore a six per cent blend was added to the design in the 2.45 minus specific gravity category with the hope of obtaining a better idea of what percentage of lightweight chert might be critical in freeze-thaw deterioration. Two beams were made for each cell of the design, i.e., two beams for each blend of each gravity group from each source.

This experiment design in the form of a three-way crossed classification allowed for statistical analysis of variance to determine the significance of the three variables in regard to the durability of concrete in which these variables were included.

Experiment Design for Shale Freeze-Thaw Study

An experimental outline was formulated in which two variables were introduced into design of the concrete beams containing shale with all controlled factors remaining constant. This experimental design differed from that of the chert study in that no specific gravity separation of the shale was made. It was set up as a two-way crossed classification. The two variables in the design were:

(a) Source of shale. Material from each of the sources was blended with the crushed stone coarse aggregate in different beams.

(b) Percentage of shale. Shale from each of the sources was combined with the crushed stone coarse aggregate in blends of two, four, six, and ten per cent.

Preparation of Concrete Test Specimens

A standard mix was designed using crushed limestone coarse aggregate (source 67-2S for the shale beams, source 53-2S for the chert beams), fine aggregate from source 79-1G, and Type I portland cement (Laboratory No. 315). The mix was designed with the Goldbeck and Gray version (15) of the b/b_o method. The mix design was held constant for all beams made except that varying small percentages of chert or shale were substituted for part of the crushed limestone coarse aggregate in all but a few control beams. A water-cement ratio of 0.46 by weight was used for all beams. The water-cement ratio produced a mix with good workability and a slump of about three inches.

The cement factor was kept constant at six bags per cubic yard. Darex was used to entrain approximately four per cent of air in each batch. Air contents were determined gravimetrically on the fresh concrete, using a 0.1 cubic- foot measure for the unit weight test.

In all beams the coarse aggregate was used in equal amounts of the #4 to 3/8-, 3/8 to 1/2-, 1/2 to 3/4-, and 3/4 to 1-inch sizes. Chert or shale was substituted for the 3/8 to 1/2-, 1/2 to 3/4-, and 3/4 to 1-inch crushed stone in two, four, six, and ten per cent blends in all but a few control beams in which the coarse aggregate consisted of 100 per cent crushed limestone. In the beams containing the deleterious materials, no deleterious materials in the #4 to 3/8-inch size range were substituted for the crushed limestone because a previous study by Sweet (59) has shown that deleterious particles passing a 3/8-inch screen have little effect on the freeze-thaw durability of concrete. He found that chert #4 to 3/8-inch in size was markedly more resistant to freeze-thaw deterioration than material larger than the 3/8-inch sieve. Sweet also found that variations in size between 3/8 inch and 1 inch did not appreciably affect the durability of concrete containing these materials, although, as would be expected, there was a slight tendency for the larger pieces within this range to be the most destructive.

As previously mentioned, each beam was batched and mixed separately to assure the correct percentage of deleterious materials in each beam. The 3/8 to 1/2- and 1/2 to 3/4-inch fractions of the deleterious materials were added to the crushed stone coarse aggregate during the batching procedure and were mixed in with the other materials

during the mixing operation to ensure random placement. In the case of the 3/4 to 1-inch deleterious material, only one piece of chert or shale was generally needed per beam for the two per cent blends and five to seven pieces for the ten per cent blends; therefore it was decided not to rely on random positioning of these pieces. Instead the 3/4 to 1-inch pieces of chert were not added to the mix until after pouring the first of the two layers into the beam molds. These pieces were then equally spaced in a row down the center of each beam where they could be expected to exert the utmost effect in causing deep-seated failure.

All the coarse aggregate was vacuum saturated before mixing. This was accomplished by means of a sealed chamber connected by individual valves to (a) a manometer, (b) a container of water free of air bubbles, and (c) a vacuum pump of the water-jet aspirator type. Dry coarse aggregate was placed in the chamber and evacuated at a pressure of approximately 2.3 cm. of mercury for a period of one hour. At the end of this period, water free of air bubbles was admitted to the chamber with the vacuum still maintained. The aggregate was covered with water and left in an immersed condition for 23 hours. The fine aggregate was not vacuum saturated, but was mixed with enough water to fill all surface-connected pores and left in this condition for 24 hours before mixing.

Mixing was accomplished by means of a modified Hobart food mixer with 1/4-cubic-foot capacity. The concrete was molded into 3- by 4- by 16-inch beams, and these beams were cured by immersion in lime water for 13 days following removal of the specimens from the molds one day after casting.

Description of Tests on Concrete Specimens

Description of Freeze-Thaw Test

The subjection of beams containing small percentages of cherts and shales to cycles of freezing and thawing was the most important part of the entire investigation. All freeze-thaw testing was done on concrete beams containing these deleterious materials; none was attempted on unconfined cherts and shales. The freeze-thaw test was conducted according to the ASTM Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water, ASTM Designation: C291-57T (1). This is a method commonly used in recent research of this type. The freezing-and-thawing apparatus consisted of an automatic-cycling freeze-thaw machine produced by the Carrier Division of the United Clay Products Company, Washington, D. C. This machine was operated at a capacity of sixty 3- by 4- by 16-inch concrete beams placed on end. The cycle length used was three and one-half hours. During the freezing phase of the cycle in which the beams were surrounded by air, the temperature at the centers of all beams was reduced from $40° \pm 3°$F. to $0° \pm 3°$F. in two hours and 45 minutes. During the thawing phase, in which the beams were surrounded by water at a temperature of $40° \pm 3°$F., the temperature at the centers of all beams was raised from $0° \pm 3°$F. to $40° \pm 3°$F. in 30 minutes. The remaining 15 minutes of the cycle was required for draining the thaw water from the freeze-thaw tank. Temperatures within the beams were checked by means of special control beams having thermistors embedded at their centers.

The end point for exposure adopted for this series of tests was 50 per cent relative dynamic modulus of elasticity or 300 cycles of freezing and thawing, whichever occurred first.

Determination of Relative Dynamic Modulus of Elasticity of Each Specimen

The fundamental transverse frequency of each beam was determined every 10-20 cycles of freezing and thawing by means of the sonic apparatus described in the ASTM Method of Test for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens, ASTM Designation: C215-58T (1). This method of test is intended primarily for detecting significant changes in the dynamic modulus of elasticity of laboratory or field test specimens that are undergoing exposure to weathering or similar deteriorating influences.

A relative dynamic modulus of elasticity, which is directly related to the strength of the specimen, was calculated for each of the fundamental transverse frequency readings by means of the following equation from ASTM Designation: C 291-57T, Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (1):

$$P_c = \frac{n_1^2}{n^2} \times 100$$

where

P_c = relative modulus of elasticity, per cent, after c cycles of freezing and thawing,

n = fundamental transverse frequency at 0 cycles of freezing and thawing, and

n_1 = fundamental transverse frequency after c cycles freezing and thawing.

Using the data obtained from the freeze-thaw testing, curves were plotted for each group of beams relating relative dynamic moduli of elasticity to cycles of freezing and thawing.

Determination of Durability Factors

A durability factor was determined for each beam according to the ASTM Method of Test for Resistance of Concrete Beams to Rapid Freezing in Air and Thawing in Water, ASTM Designation: C 291-57T (1). The durability factor was calculated as follows:

$$DF = \frac{PN}{M}$$

where

DF = durability factor of the test beam,

P = relative dynamic modulus of elasticity at N cycles, per cent,

N = number of cycles at which P reaches the specified minimum value for discontinuing the test (relative E = 50 per cent), or the specified number of cycles at which exposure is to be terminated (300 cycles),whichever is less, and,

M = specified number of cycles (300) at which the exposure is to be terminated.

This durability factor is of importance as an index of the relative freeze-thaw resistance of concrete beams made with different

variables. After the durability factors were determined for all the beams tested, they were subjected to statistical analysis wherever applicable to determine the significance of the effects of the variables on the durability of the concrete.

Study of Surface Deterioration of the Specimens

During the freeze-thaw testing program, a number of popouts occurred on the surfaces of some of both the chert and shale beams. These popouts often occurred on beams which showed no deep-seated failure such as would be evidenced by low durability factors. Since these popouts were all found to be caused by failure of pieces of chert and shale during the freeze-thaw testing, this was further studied. During the freeze-thaw testing program, visual observation of any new popouts was made each time a beam was tested for its fundamental transverse frequency, i.e., every 10-20 cycles of freezing and thawing. The position of each popout was noted as well as the approximate size of the piece of deleterious material causing it.

In order to compare the relative severity of surface deterioration of the beams, it was necessary to determine an index number for each beam which would give an indication of the relative popout damage suffered by that beam. An arbitrary numerical index based on sizes of the deleterious particles causing the popouts, number of popouts, and numbers of cycles at which the popouts occurred was developed and can be explained as follows:

$$\text{SDF} = \sum \left[\frac{s_1}{c_1} + \frac{s_2}{c_2} + \frac{s_3}{c_3} + \ldots\ldots + \frac{s_n}{c_n} \right]$$

where

SDF = surface durability factor for each beam,

s = size factor, 1 for popouts caused by deleterious particles 3/8 to 1/2 inch in size, 2 for popouts 1/2 to 3/4-inch in size (average of 2 diameters),

c_1 = cycle factor, 1 for cycles 1 to 100, 2 for cycles 101 to 200, 3 for cycles 201 to 300.

For beams whose relative moduli of elasticity dropped below 50 before 300 cycles of freezing and thawing were attained, this arbitrary equation does not result in an index that can be compared with beams undergoing the full 300 cycles. In many cases the beams that were removed from the freeze-thaw test before 300 cycles would have suffered additional surface deterioration if they had been allowed to reach 300 cycles. It is difficult to devise a correction factor which would satisfactorily eliminate this failing of the equation. However, only a few of the beams tested fall in this category, and these were especially noted in tabulating the data so that no direct comparisons would be made.

Study of Air Voids in Concrete by Linear Traverse Technique

During the early stages of the freeze-thaw study of concrete beams containing shales, a few beams were prepared from hand-mixed concrete to see how their durabilities would compare with the regular beams from machine-mixed concrete used in the freeze-thaw testing program. It was found that in nearly all cases the hand-mixed concrete had lower durability factors than machine-mixed concrete

containing the same amounts of the same materials. In an effort to determine the reason for this notable difference in durability, a subsequent study was made of the character of the air voids in several of the beams by means of the linear traverse technique outlined by Fears (12). It was felt that a comparison of air bubble size and spacing for the hand-mixed and machine-mixed concretes might provide laboratory proof of the theoretical concept developed by Powers (40).

Powers theorized that the increase in durability afforded concrete by means of air entrainment is largely a function of the spacing of the air voids in the concrete. He suggested that a concrete containing air voids with high specific surface area, and thus with many small voids spaced close together, would receive more protection from the air voids than one with a low specific surface area and with the voids spaced relatively far apart.

The air-void study was conducted by means of the linear traverse technique (12). For this study pairs of beams containing shale were chosen in which the concrete in both beams had been made from identical blends, but one was hand-mixed and the other was machine-mixed. In all cases the hand-mixed concrete had resulted in lower durability factors than the machine-mixed concrete. By means of the linear traverse technique, the total percentage of entrained and entrapped air as well as the bubble spacing was determined for each beam. As recommended by Fears,a 200-inch traverse, consisting of 20 equally spaced 10-inch paths for each beam was run on polished surfaces of two slabs cut from each beam. Using the values obtained for total percentage of air and voids per inch of traverse, it was possible to

calculate specific surface areas and void spacing factors for the beams according to the method developed by Powers (40). The specific surface areas were computed as follows:

$$\alpha = \frac{4n}{A}$$

where

$\alpha =$ specific surface area of voids in sq.in./cu.in.,

$n =$ voids per inch of traverse,

$A =$ total air content of the beam in per cent.

The void spacing factors were computed as follows:

$$L = r_h - r_m$$

where

$L =$ spacing factor in inches,

$r_h =$ radius in inches of hypothetical sphere $= \frac{3}{\alpha}$,

$r_m =$ radius in inches of sphere of influence

$$= \frac{\sqrt{3}}{2}\left(\frac{p + A}{N}\right)^{1/3} .$$

$p =$ paste content per unit volume of mix,

$A =$ total air content in per cent,

$N =$ hypothetical number of voids per cubic inch

$$= \frac{A\,\alpha^3}{36\,\pi}$$

Powers considered a void spacing factor of 0.01 inches to be critical; those concretes with spacing factors lower than 0.01 inches

were thought to be well protected from freezing and thawing deterioration; those with spacing factors greater than 0.01 inches were thought to be poorly protected.

This study was conducted on seven pairs of shale beams to attempt to determine a laboratory relationship between the air-void spacing factor of air-entrained concrete containing deleterious aggregates and the durability of this concrete. It was intended as a laboratory check of Powers' theoretical choice of 0.01 inches as a critical value of the void spacing factor.

RESULTS OF TESTS OF BASIC PROPERTIES OF CHERTS AND SHALES

The results of tests of basic properties of the cherts and shales have been divided into the following sections: (a) specific gravity tests, (b) porosity determinations, (c) absorption tests.

Specific Gravity Tests

Fractionation by Heavy Liquids

Heavy liquid separation was performed on each of the six cherts listed in Table 1 and the five shales listed in Table 2 to determine the specific gravity distribution (bulk saturated surface-dry) within each source. The results of the specific gravity separations of the cherts are shown in Table 3 and in Figure 5. The results of the shale separations are presented in Table 4 and in Figure 6.

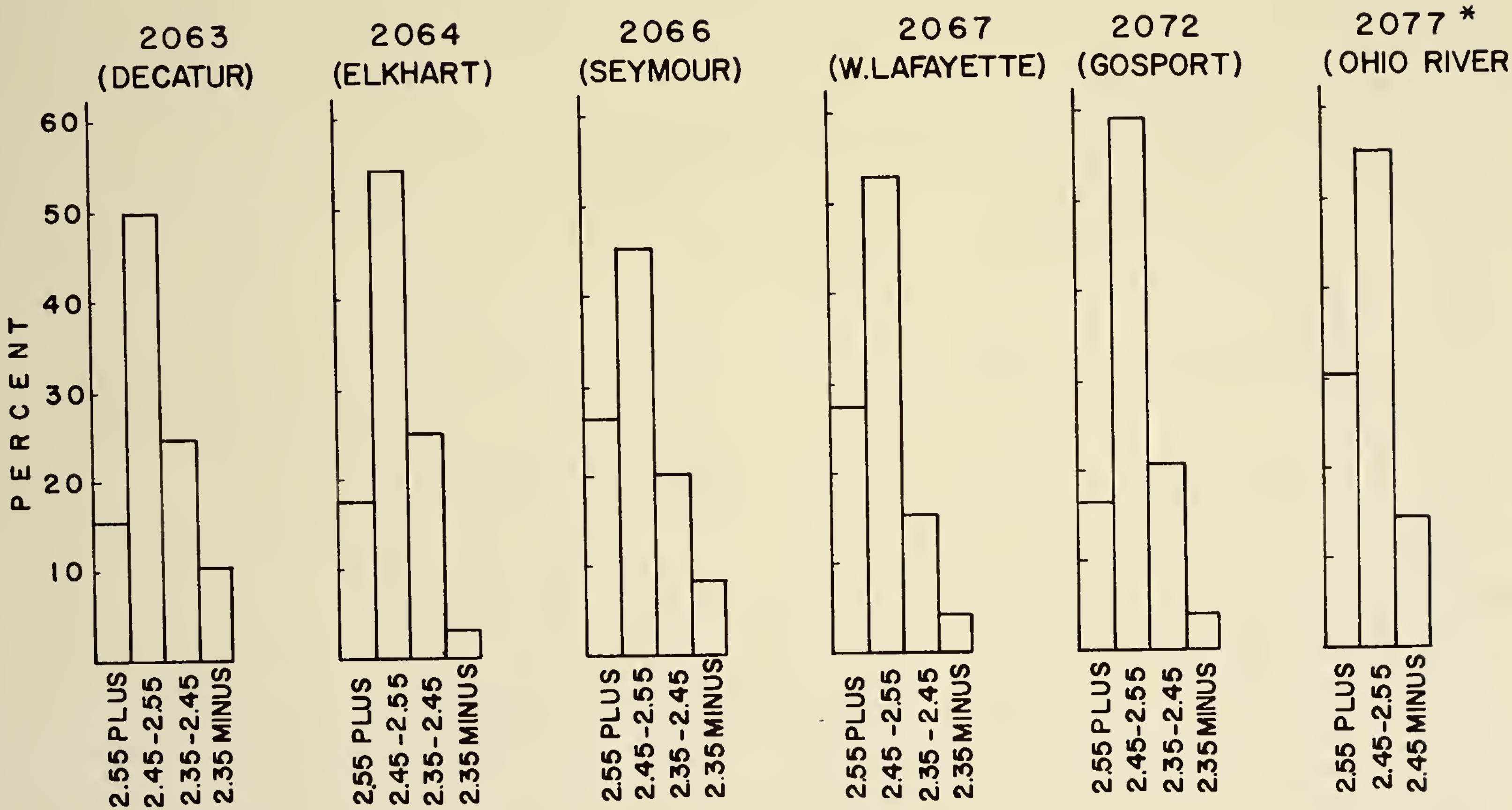
Each sample was divided into size groups before separation, and mean percentages in the various specific gravity fractions were determined for each sample by averaging the percentages obtained for each size group in the sample. For example, for chert 2063, 2.55 plus specific gravity material comprised 22.1 per cent of the 3/4 to 1-inch size group, 12.4 per cent of the 1/2 to 3/4-inch group, and 11.6 per cent of the 3/8 to 1/2-inch group. Therefore the mean percentage of material in the 2.55 plus gravity group is the 15.4 per cent

Table 3

Percentages of Chert Samples in Bulk Specific Gravity Ranges (Saturated Surface-Dry Basis) as Determined by Heavy Liquid Separation

Sample Number	Size Range (inches)	Percentage in Each Specific Gravity Range			
		2.55 plus	2.45-2.55	2.35-2.45	2.35 minus
2063 (Decatur)	3/4-1	22.1	46.6	18.6	12.6
	1/2-3/4	12.4	48.6	29.0	9.7
	3/8-1/2	11.6	54.8	25.4	8.3
	Average	15.4	50.0	24.3	10.2
2064 (Elkhart)	3/4-1	21.7	54.2	24.1	0.0
	1/2-3/4	17.1	61.0	18.6	3.3
	3/8-1/2	13.8	47.9	32.5	5.8
	Average	17.5	54.4	25.1	3.0
2066 (Seymour)	3/4-1	24.8	50.1	17.9	7.2
	1/2-3/4	24.4	41.7	24.1	9.8
	3/8-1/2	29.3	44.0	18.7	8.0
	Average	26.2	45.3	20.2	8.3
2067 (West Lafayette)	3/4-1	23.9	56.8	13.7	5.5
	1/2-3/4	28.3	56.2	12.5	3.0
	3/8-1/2	29.3	46.4	19.6	4.7
	Average	27.2	53.2	15.3	4.4
2072 (Gosport)	3/4-1	16.7	67.3	15.0	1.0
	1/2-3/4	14.3	60.2	22.7	2.8
	3/8-1/2	18.1	49.7	25.0	7.1
	Average	16.4	59.1	20.9	3.6
				2.45 minus	
2077* (Ohio River)	3/4-1	36.7	49.6	13.7	
	1/2-3/4	25.4	61.7	12.9	
	3/8-1/2	29.2	55.0	15.8	
	Average	30.4	55.4	14.2	

*Not separated at 2.35 level



* NOT SEPARATED AT 2.35 LEVEL.

FIGURE 5. PERCENTAGES OF CHERT SAMPLES IN DIFFERENT BULK SPECIFIC GRAVITY RANGES (SATURATED SURFACE-DRY BASIS)

Table 4

Percentages of Shale Samples in Bulk Specific Gravity Ranges (Saturated Surface-Dry Basis) as Determined by Heavy Liquid Separation

Sample Number	Size Range (inches)	Percentage in Each Specific Gravity Range						
		2.55 Plus	2.45-2.55	2.35-2.45	2.25-2.35	2.15-2.25	2.05-2.15	2.05 minus
2063 (Decatur)	3/4-1	2.0	7.7	22.2	39.4	13.2	15.4	0.0
	1/2-3/4	2.8	3.2	39.8	29.5	13.6	6.8	4.2
	3/8-1/2	6.8	8.8	21.6	40.3	13.3	4.3	4.9
	Average	3.9	6.6	27.9	36.4	13.4	8.8	3.0
2066 (Seymour)	3/4-1	0.0	0.0	1.6	7.3	23.0	36.7	31.4
	1/2-3/4	0.0	0.0	0.0	9.2	22.2	41.0	27.6
	3/8-1/2	0.0	0.0	1.2	5.5	30.5	31.5	31.3
	Average	0.0	0.0	0.9	7.3	25.2	36.4	30.1
2068 (near La Porte)	3/4-1	0.0	0.0	0.0	0.2	56.6	43.2	0.0
	1/2-3/4	0.0	0.0	0.9	1.7	65.3	29.6	2.7
	3/8-1/2	0.0	0.0	0.0	2.0	47.3	46.2	4.5
	Average	0.0	0.0	0.3	1.3	56.4	39.7	2.4
2075 (Indianapolis)	3/4-1	1.1	4.1	29.7	34.7	24.6	3.6	1.9
	1/2-3/4	2.1	2.1	26.2	45.2	15.1	6.3	2.9
	3/8-1/2	1.7	2.4	22.4	44.8	18.6	8.3	1.9
	Average	1.6	2.9	26.1	41.6	19.4	6.1	2.2
2076 (South Bend)	3/4-1	0.0	0.0	0.0	12.4	76.5	11.1	0.0
	1/2-3/4	0.0	0.0	0.0	5.7	68.3	26.0	0.0
	3/8-1/2	0.0	0.0	0.0	18.4	57.1	24.3	0.2
	Average	0.0	0.0	0.0	12.2	67.3	20.5	0.1

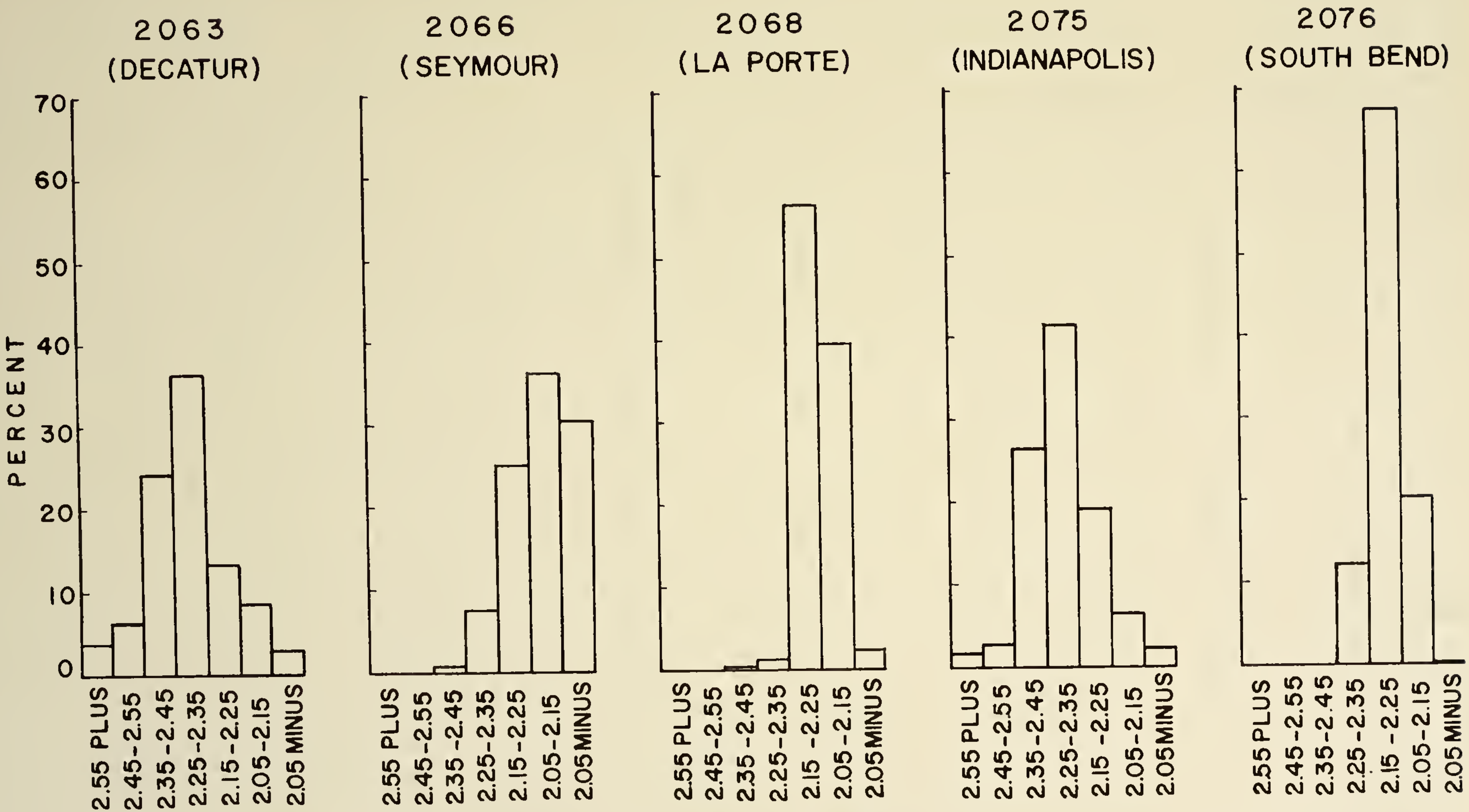


FIGURE 6. PERCENTAGES OF SHALE SAMPLES IN DIFFERENT BULK SPECIFIC GRAVITY RANGES (SATURATED SURFACE-DRY BASIS)

obtained by averaging these three values. The mean percentage obtained in this way assumes an equal amount of material in each size range as was the case when the cherts and shales were used in concrete beams for freeze-thaw study.

Determination of Bulk Specific Gravity

Bulk specific gravity values were determined for the chert and shale samples primarily for use in calculating the total porosities of these materials. In addition,bulk specific gravities on saturated surface-dry basis were determined as a check on the values obtained by heavy-liquid separation.

The bulk specific gravity values were obtained using a modification of ASTM Designation: C127-42, Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregate (1).For the cherts, samples were tested from the 2.55 plus, 2.45-2.55, and 2.45 minus bulk specific gravity ranges (saturated surface-dry basis) of cherts 2067 and 2077. These samples had been obtained by heavy-liquid separation. For the shales, the samples tested for each source were from a bulk specific gravity range (saturated surface-dry basis) very close to the average specific gravity for the original sample from each source obtained by heavy-liquid separation.

The bulk specific gravity values for the cherts determined by this method are shown in Table 5. Those for the shales are in Table 6. Two tests were run on each sample, and in all cases the two values checked closely. The average value for each sample is shown in these tables.

Table 5

Bulk Specific Gravity Values for Cherts, Obtained by a Modification of ASTM Designation: C127-42, Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregates

Source	Specific Gravity Range (by heavy-liquid separation)	Bulk Specific Gravity	Bulk Specific Gravity (saturated surface-dry basis)
2067 (West Lafayette)	2.55 plus	2.56	2.58
	2.45-255	2.44	2.50
	2.45 minus	2.30	2.41
2077 (Ohio River)	2.55 plus	2.56	2.58
	2.45-2.55	2.47	2.51
	2.45 minus	2.31	2.41

Table 6

Bulk Specific Gravity Values for Shales, Obtained by a Modification of ASTM Designation: C127-42, Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregates

Source	Specific Gravity Range (by heavy-liquid separation)	Bulk Specific Gravity	Bulk Specific Gravity (saturated surface-dry basis)
2063 (Decatur)	2.30-2.32	2.28	2.31
2066 (Seymour)	2.13-2.15	2.06	2.18
2068 (near La Porte)	2.17-2.19	2.00	2.22
2075 (Indianapolis)	2.29-2.32	2.24	2.32
2076 (South Bend)	2.19-2.21	2.08	2.23

Determination of True Specific Gravity

True specific gravity was determined by the pycnometer method for each of the three specific gravity groups from chert samples 2067 and 2077, and for each of the average specific gravity samples obtained from shales from the five shale sources. Each of the chert samples was tested once. In each case the value obtained was very close to the true specific gravity of quartz (2.65) which is the theoretical true specific gravity of a pure chert. Of the tests run on the five shale samples, the two resulting in the extreme (the highest and lowest) specific gravity values were repeated, and in both cases duplicate results were obtained. therefore it is assumed that all the values obtained are reliable.

The results of the pycnometer tests of the chert samples are shown in Table 7. The true specific gravities for the shales are presented in Table 8.

Porosity Determinations

Total Porosity

The total porosity or ratio of total voids to bulk volume of the aggregate was calculated for each of the three specific gravity groups from chert sources 2067 and 2077, and for each of the five shale sources. These values were determined by use of the bulk specific gravity and true specific gravity values for each sample.

The total porosities of the chert samples are shown in Table 9. The total porosities of the shales are presented in Table 10.

Table 7

True Specific Gravity Values for Chert Samples

Source	Specific Gravity Group (by heavy-liquid separation)		
	2.55 plus	2.45-2.55	2.45 minus
2067 (West Lafayette)	2.64	2.64	2.64
2077 (Ohio River)	2.64	2.64	2.65

Table 8

True Specific Gravity Values for Shale Samples

Source	True Specific Gravity
2063 (Decatur)	2.38
2066 (Seymour)	2.39
2068 (near La Porte)	2.58
2075 (Indianapolis)	2.45
2076 (South Bend)	2.47

Table 9

Total Porosities of Chert Samples

Source	Gravity Group (by heavy liquid (separation)	Bulk Sp.Gr.	True Sp.Gr.	$\frac{S_b}{S_t}$ *	Porosity, n (per cent)
2067 (West Lafayette)	2.55+	2.56	2.64	0.970	3.0
	2.45-2.55	2.44	2.64	0.924	7.6
	2.45 minus	2.30	2.64	0.871	12.9
2077 (Ohio River)	2.55+	2.56	2.64	0.970	3.0
	2.45-2.55	2.47	2.64	0.936	6.4
	2.45 minus	2.31	2.65	0.872	12.8

* $n = (1 - \frac{S_b}{S_t})100$

Table 10

Total Porosities of Shale Samples

Source	Bulk Specific Gravity	True Specific Gravity	$\frac{S_b}{S_t}$*	Porosity, n (per cent)
2063 (Decatur)	2.28	2.38	0.958	4.2
2066 (Seymour)	2.06	2.39	0.862	13.8
2068 (near La Porte)	2.00	2.58	0.775	22.5
2075 (Indianapolis)	2.24	2.45	0.914	8.6
2076 (South Bend)	2.08	2.47	0.842	15.8

* $n = (1 - \frac{S_b}{S_t})100$

Size-Studies of Pores

The percentage of total volume consisting of voids less than 5 microns in diameter (microvoids) was calculated by subtracting the percentage of total volume of voids greater than 5 microns in diameter, obtained by microscopic study of polished sections, from the total porosity of the sample. The percentages of voids less than 5 microns in diameter are presented in Table 11 for the three specific gravity groups of chert samples 2067 and 2077. This table also shows the results of the microscopic linear traverse studies given as percentages of total volume consisting of voids greater than 5 microns in diameter. No linear traverse studies were run on the shales because they could not be polished well enough to discern the small voids. A sample data sheet for the linear traverse studies is included in Table 30 in Appendix C.

Absorption Tests

Vacuum-Saturated Absorption

For the vacuum-saturated absorption tests the dry aggregates were evacuated for one hour in a sealed chamber, immersed while still in the evacuated state, and left in an immersed condition for 23 hours. The results of these tests on 54 chert samples (three specific gravity groups from each of three size ranges for six chert sources) are shown in Table 12. Table 13 presents the vacuum-saturated absorption values for 15 shale samples (three size ranges for each of the five shale sources).

Table 11
Per Cent Voids Greater Than and Less Than 5 Microns in Diameter for the Chert Samples

Source	Gravity Group	Polished Section Number	Per Cent Voids >5 Microns in Diameter	Porosity, n (per cent)	Per Cent Voids <5 Microns in Diameter
2067 (West Lafayette)	2.55 plus	1	0.46		
		2	1.06		
		3	0.37		
		Avg.	0.63	3.0	2.4
	2.45-2.55	1	1.13		
		2	2.38		
		3	2.08		
		Avg.	1.86	7.6	5.7
	2.45 minus	1	4.56		
		2	10.92		
		3	3.98		
		Avg.	6.49	12.9	6.4
2077 (Ohio River)	2.55 plus	1	0.56		
		2	0.32		
		3	0.23		
		Avg.	0.37	3.0	2.6
	2.45-2.55	1	1.80		
		2	1.68		
		3	3.50		
		Avg.	2.33	6.4	4.1
	2.45 minus	1	11.75		
		2	4.46		
		3	7.46		
		Avg.	7.89	12.8	4.9

Table 12

Vacuum-Saturated Absorption Values for Chert Samplea

Source	Saturated Surface-Dry Bulk Specific Gravity Group	Size Range (inches)	Absorption (per cent)
2063 (Decatur)	2.55 plus	3/4-1	1.26
		1/2-3/4	1.21
		3/8-1/2	1.12
			Avg. 1.20
	2.45-2.55	3/4-1	3.02
		1/2-3/4	2.84
		3/8-1/2	3.30
			Avg. 3.05
	2.45 minus	3/4-1	6.26
		1/2-3/4	6.13
		3/8-1/2	6.88
			Avg. 6.42
2064 (Elkhart)	2.55 plus	3/4-1	0.82
		1/2-3/4	0.93
		3/8-1/2	1.00
			Avg. 0.92
	2.45-2.55	3/4-1	2.86
		1/2-3/4	2.78
		3/8-1/2	2.90
			Avg. 2.85
	2.45 minus	3/4-1	5.71
		1/2-3/4	5.56
		3/8-1/2	5.62
			Avg. 5.63

Table 12 (continued)

Vacuum-Saturated Absorption Values for Chert Samples

Source	Saturated Surface-Dry Bulk Specific Gravity Group	Size Range (inches)	Absorption (per cent)
2066 (Seymour)	2.55 plus	3/4-1	1.14
		1/2-3/4	1.30
		3/8-1/2	1.09
			Avg. 1.18
	2.45-2.55	3/4-1	2.76
		1/2-3/4	3.12
		3/8-1/2	2.86
			Avg. 2.91
	2.45 minus	3/4-1	6.12
		1/2-3/4	6.36
		3/8-1/2	6.11
			Avg. 6.20
2067 (West Lafayette)	2.55 plus	3/4-1	1.09
		1/2-3/4	1.05
		3/8-1/2	1.19
			Avg. 1.11
	2.45-2.55	3/4-1	2.96
		1/2-3/4	2.84
		3/8-1/2	2.67
			Avg. 2.82
	2.45 minus	3/4-1	5.58
		1/2-3/4	5.59
		3/8-1/2	5.62
			Avg. 5.60

Table 12 (continued)

Vacuum-Saturated Absorption Values for Chert Samples

Source	Saturated Surface-Dry Bulk Specific Gravity	Size Range (inches)	Absorption (per cent)
2072 (Gosport)	2.55 plus	3/4-1	1.02
		1/2-3/4	1.11
		3/8-1/2	1.11
			Avg. 1.08
	2.45-2.55	3/4-1	2.55
		1/2-3/4	2.80
		3/8-1/2	2.96
			Avg. 2.77
	2.45 minus	3/4-1	5.26
		1/2-3/4	5.39
		3/8-1/2	5.91
			Avg. 5.52
2077 (Ohio River)	2.55 plus	3/4-1	1.06
		1/2-3/4	0.92
		3/8-1/2	1.04
			Avg. 1.01
	2.45-2.55	3/4-1	2.31
		1/2-3/4	2.69
		3/8-1/2	2.27
			Avg. 2.42
	2.45 minus	3/4-1	4.77
		1/2-3/4	4.20
		3/8-1/2	4.51
			Avg. 4.49

Table 13

Vacuum-Saturated Absorption Values for Shale Samples

Source	Size Range (inches)	Absorption (per cent)
2063 (Decatur)	3/4-1	1.54
	1/2-3/4	1.60
	3/8-1/2	2.17
		Avg. 1.77
2066 (Seymour)	3/4-1	7.07
	1/2-3/4	7.05
	3/8-1/2	7.04
		Avg. 7.05
2068 (near La Porte)	3/4-1	12.83
	1/2-3/4	12.23
	3/8-1/2	12.63
		Avg. 12.56
2075 (Indianapolis)	3/4-1	3.64
	1/2-3/4	4.06
	3/8-1/2	4.68
		Avg. 4.13
2076 (South Bend)	3/4-1	7.82
	1/2-3/4	8.61
	3/8-1/2	7.91
		Avg. 8.11

Table 14 presents the vacuum-saturated surface-dry absorption values for broken chert pebbles taken from deep seated cracks in beams made from each of the chert sources. Each absorption value represents a test on the broken pieces of a single pebble which appeared to be the causal factor in the formation of a deep-seated crack. Each of these pebbles belonged to the 2.45 minus saturated surface-dry bulk specific gravity group.

Rate of Absorption at Atmospheric Pressure

The results of the rate of absorption tests on the three gravity groups from chert samples 2067 and 2077 which were subjected to most of the tests of basic properties are presented graphically on a semi-logarithmic plot of time versus per cent absorption in Figure 7. The same type information is shown for the five shale samples in Figure 8.

Petrographic Studies

These studies included microscopic examination of thin sections, and x-ray and differential thermal analyses to determine the mineral compositions and textural and microstructural features which might influence the freeze-thaw durabilities of the cherts and shales.

Petrographic analysis of thin sections showed the cherts to be of generally similar mineralogical character. They are composed primarily of microcrystalline quartz and radial chalcedony. Small amounts of coarser-grained secondary quartz, some calcite, and limonite and carbonate rhombs are also present. One chief mineralogical difference was noted. Cherts from the southern part of Indiana (cherts 2066,

Table 14

Vacuum-Saturated Absorption Values for Individual Pieces of Chert Obtained from Deep-seated Cracks in Freeze-Thaw Test Beams

Beam Number	Chert Source	Saturated Surface-Dry Bulk Specific Gravity Group	Per Cent Absorption for the Individual Pieces	Average Per Cent Absorption for This Type of Material (From Table 12)
K9-2	2063 (Decatur)	2.45 minus	7.05	6.42
L9-1	2064 (Elkhart)	2.45 minus	7.81	5.63
M9-1	2066 (Seymour)	2.45 minus	9.69	6.20
09-2	2067 (West Lafayette)	2.45 minus	4.93	5.60
P9-2	2072 (Gosport)	2.45 minus	3.27	5.52
R8-1	2077 (Ohio River)	2.45 minus	7.02	4.49
R9-1	2077 (Ohio River)	2.45 minus	6.55	4.49
R9-2	2077 (Ohio River)	2.45 minus	5.99	4.49

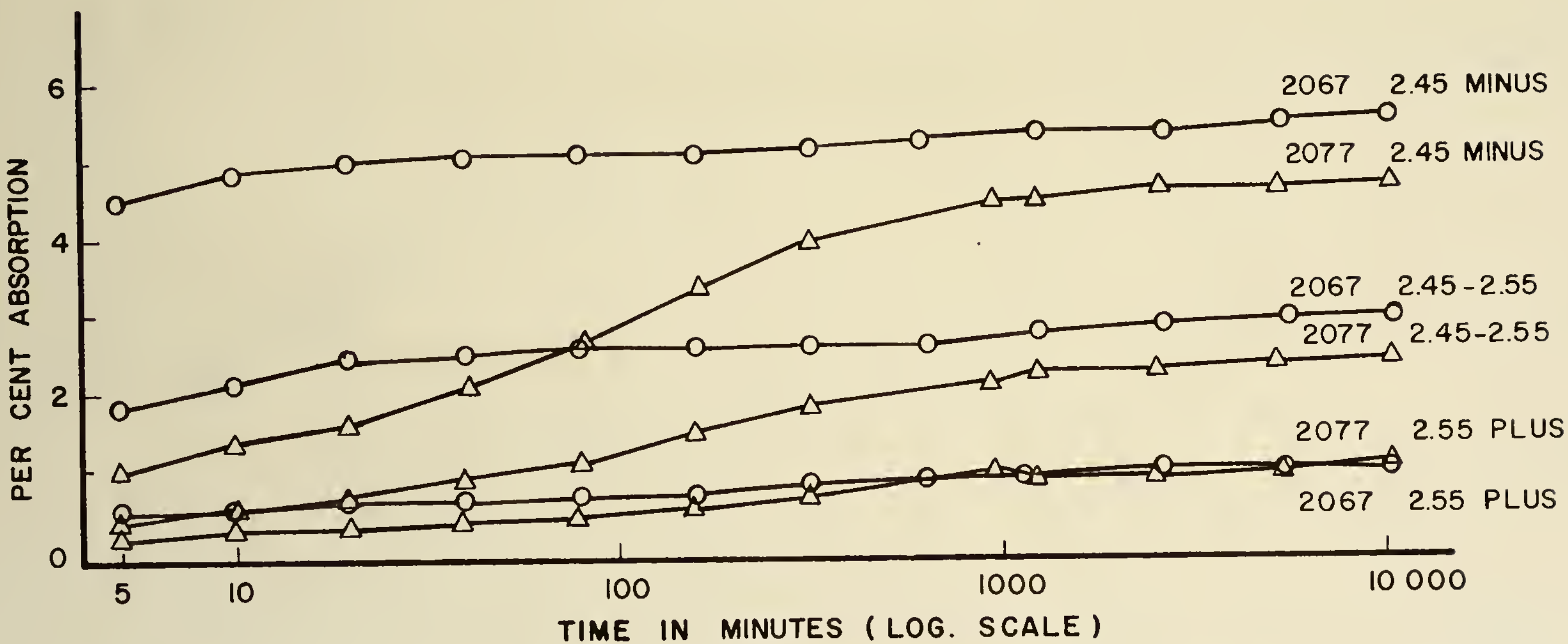


FIGURE 7. RATES OF ABSORPTION FOR DIFFERENT SPECIFIC GRAVITY FRACTIONS OF CHERTS 2067 AND 2077

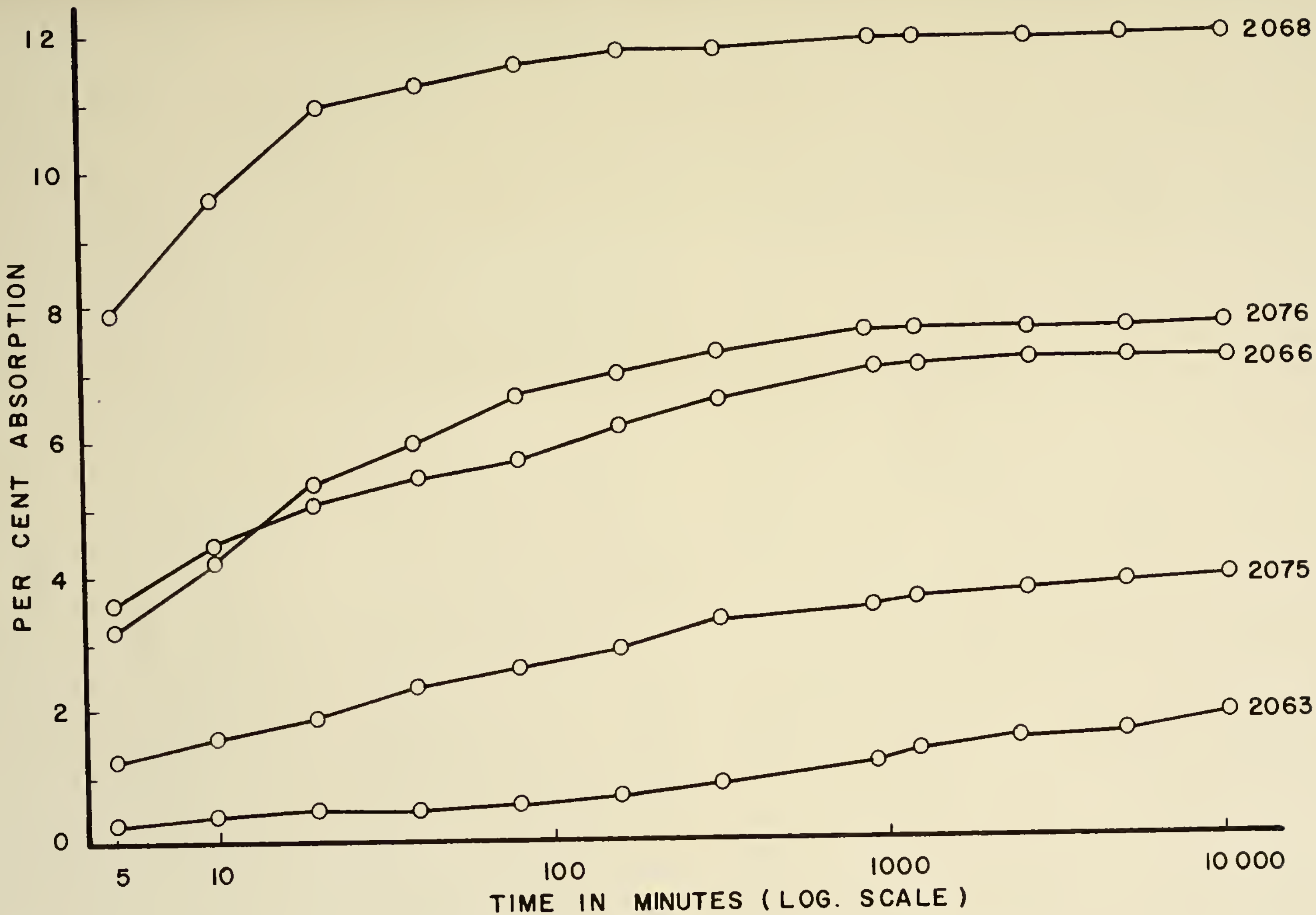


FIGURE 8. RATES OF ABSORPTION FOR SHALE SAMPLES

2072, and 2077) generally contain larger percentages of limonite and limonite rhombs than those from the northern part of the state (cherts 2063, 2064, and 2067). This is especially true for sample 2077 which is typical of the brown Ohio River cherts. An important microstructural feature in samples from all six sources is the numerous voids observed in thin sections from material in the 2.45 minus specific gravity range. No voids were noted in the 2.45-2.55 and the 2.55 plus ranges. The voids are all fairly large, since voids less than about 30 microns in diameter cannot be detected easily in thin-section study. They range in size up to 0.4-0.5 mm. in diameter, but most were less than 0.1 mm. in diameter. It seems obvious that the presence of these voids largely accounts for the low bulk specific gravity of chert in this range.

Each of the cherts consists primarily of microcrystalline aggregates of quartz grains usually less than 0.01 mm. in diameter. Secondary quartz occurs as granular masses which apparently replaced carbonate minerals. The individual quartz grains in these secondary masses range in size from less than 0.01 mm. to as large as 0.2 mm. Radiating chalcedony in the chert samples occurs as spherulites, often as much as 0.25 mm. wide.

The carbonate and limonite rhombs range in size from less than 0.01 mm. to as much as 0.1 mm. Although most of the rhombs consisted of carbonate <u>or</u> limonite, some appeared to have a translucent carbonate mineral in the center surrounded by a rim of opaque limonite. The carbonate rhombs probably formed by replacement of crystalline quartz in the original chert (68), and in turn these rhombs are being replaced

by limonite. Some of the voids occurring in the 2.45 minus specific gravity chert undoubtedly were formed by solution of carbonate rhombs without replacement of the carbonate by limonite. Limonite also occurs as finely disseminated masses scattered throughout the chert. Apparently neither the limonite rhombs nor the finely divided limonite have any effect on the freeze-thaw durability of the chert.

Petrographic, x-ray, and differential thermal analyses of the shales indicate a similarity in their general mineralogic composition, but there is considerable variation in certain characteristics. All the shales consist of detrital mineral grains, primarily quartz, in a very fine-grained matrix of clay minerals or hydromicas. The chief differences shown by the shales are the relative size and abundance of the detrital minerals and the relative amounts of clay minerals and organic material in the samples.

Microscopic petrography was used for study of detrital mineralogy, textures, and microstructures of the shales. The clay mineralogy was determined primarily by x-ray diffraction and differential thermal analysis. An example of an x-ray spectrometer trace obtained in this study is shown in Figure 20 of Appendix A. Loss on ignition tests were conducted on each of the samples to provide an indication of the amount of organic material present.

In order to satisfactorily describe the shales and to point out differences in their petrographic characteristics, and yet avoid repetition, a brief petrographic description of a shale with "average" characteristics will be given, and the mineralogies, textures, and microstructures of the strongest, least porous shale (2063) and the

weakest, most porous shale (2068) will be compared with those of the average sample. A brief summary of the results of petrographic studies of all five shales is presented in Table 23 of Appendix A.

The average shale is composed primarily of a fine-grained illite matrix enclosing detrital quartz grains up to 0.04 mm. in diameter (0.01-0.02 mm. average). It contains considerable organic matter and disseminated limonite and a small amount of chlorite. Loss on ignition for this shale is approximately 12 per cent. This description fits shales 2066, 2075, and 2076 fairly well.

Shale 2063, the most indurated and least porous of the shales, contained more detrital quartz and less clay mineral than the average shale. Besides being more abundant,the detrital quartz grains are larger than in the other shales, ranging in size up to 0.07 mm. in diameter (0.02 - 0.03 mm. average). The abundance and size of the detrital quartz is sufficient to classify sample 2063 as a silty shale or possibly even a siltstone. This sample also contained more organic material than the other shales as was shown by the 16.7 per cent loss on ignition, the highest of all the shales.

Shale 2068, the softest and most porous of the shales, contains more clay mineral and less detrital quartz than the other shales. The relatively high percentage of illite accounts for the high porosity of this shale, and the combination of high clay and low quartz content accounts for its lack of induration.

Study of thin sections, cut normal to the bedding of the shales, by means of a technique similar to that used by Mitchell on clays (36) showed little difference in the amount of preferred orientation

of clay particles for the different shales. The majority of the clay particles were oriented with their short "c" axes normal to the bedding (see Figures 17 and 18) but it was not possible to discern differences in the amount of orientation of particles in different shales as reflected in differences in maximum illumination between crossed nicols.

Figure 9

Chert 2063 (s.g. 2.55 plus) in plain light.
Carbonate rhombs in fine-grained quartz matrix.

Figure 10

Chert 2067 (s.g. 2.45-2.55) in plain light.
Large carbonate mass in center of photograph.
Note many carbonate (translucent) and limonite (opaque) rhombs in fine-grained quartz matrix.
Magnification is about one-third that of the other photographs in these figures.

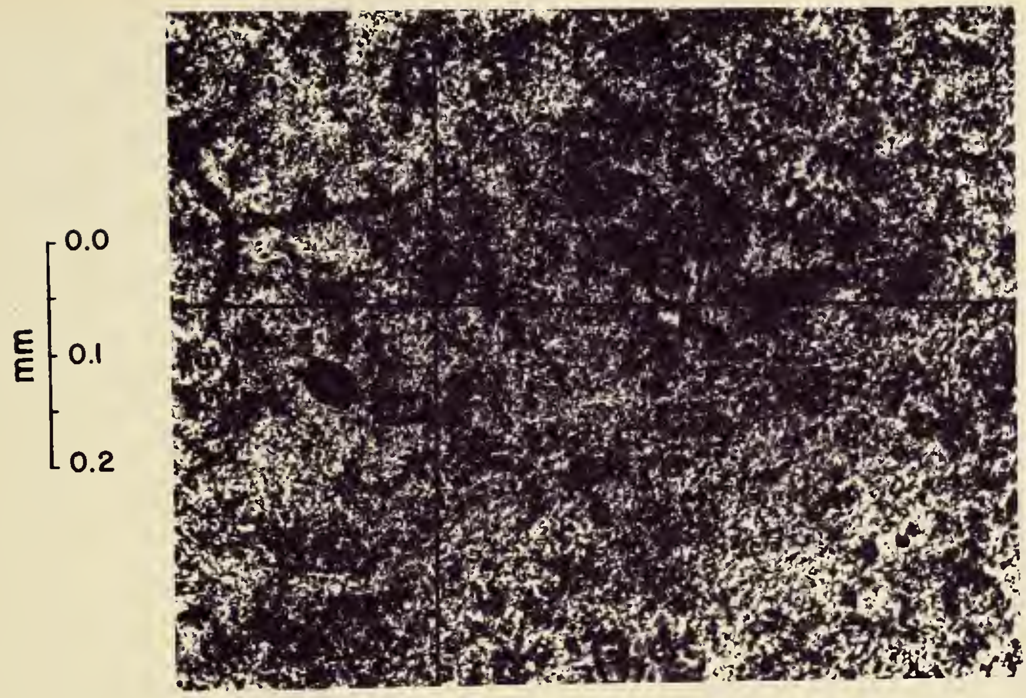


FIGURE 9.

FIGURE 10.

Figure 11

Chert 2064 (s.g. 2.45-2.55) between crossed nicols.
Large secondary quartz grains in fine-grained quartz matrix.
The secondary quartz has replaced a carbonate fossil.

Figure 12

Chert 2064 (s.g. 2.55 plus) between crossed nicols.
Radial chalcedony in fine-grained quartz matrix.

FIGURE 11.

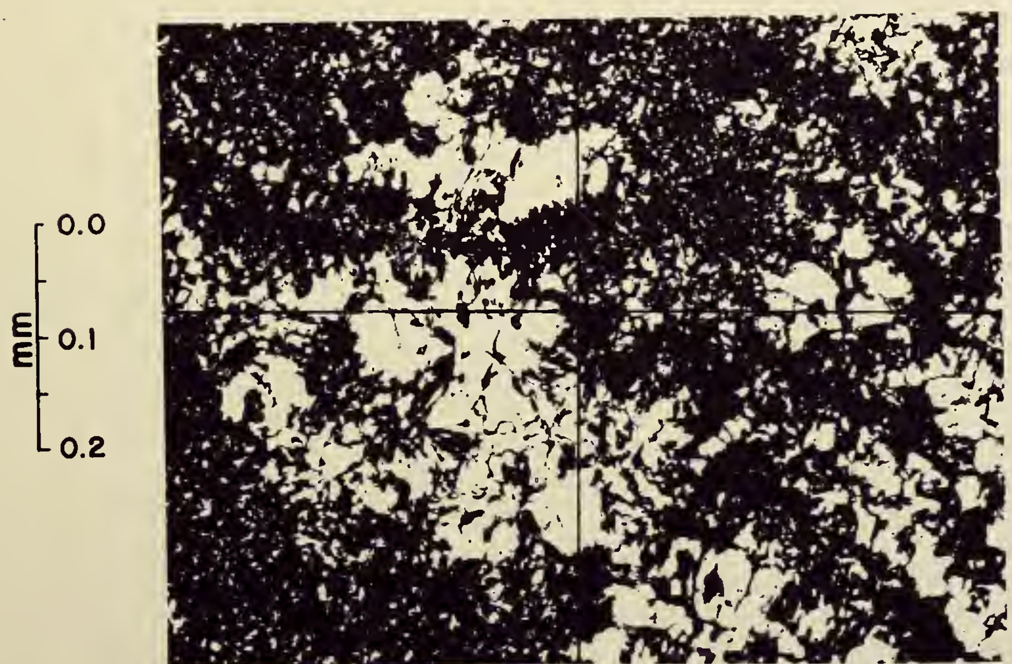


FIGURE 12.

Figure 13

Chert 2077 (s.g. 2.45 minus) in plain light.
Limonite (opaque) and carbonate (translucent rhombs in fine-grained quartz matrix.
Note void in process of formation by solution of carbonate from large rhomb near intersection of cross-hairs.

Figure 14

Chert 2063 (s.g.2.45 minus) between crossed nicols.
Large voids surrounded by radial chalcedony and fine-grained quartz matrix.

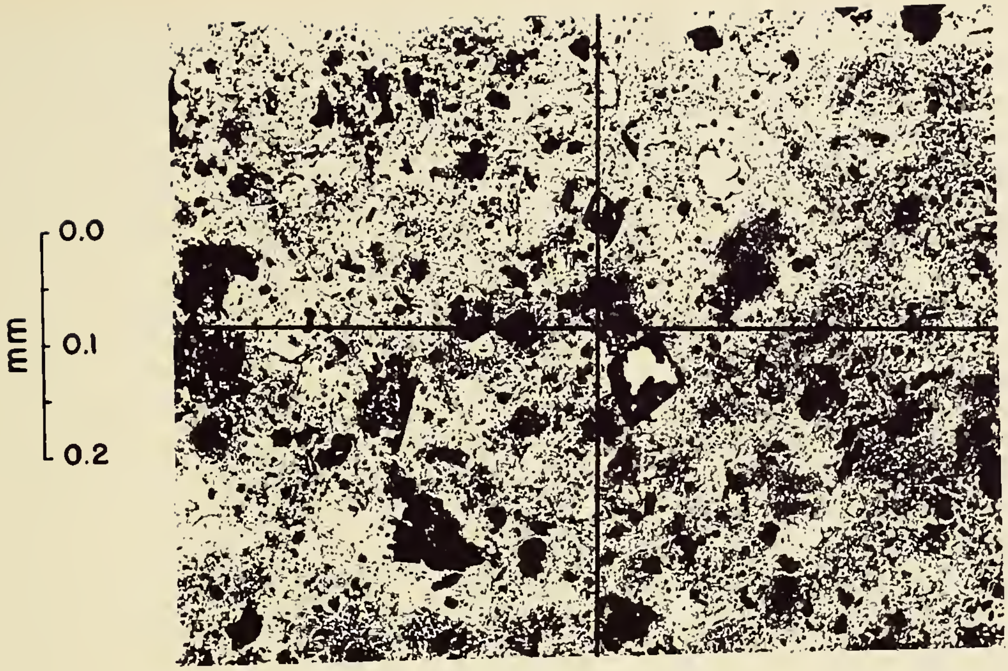


FIGURE 13.

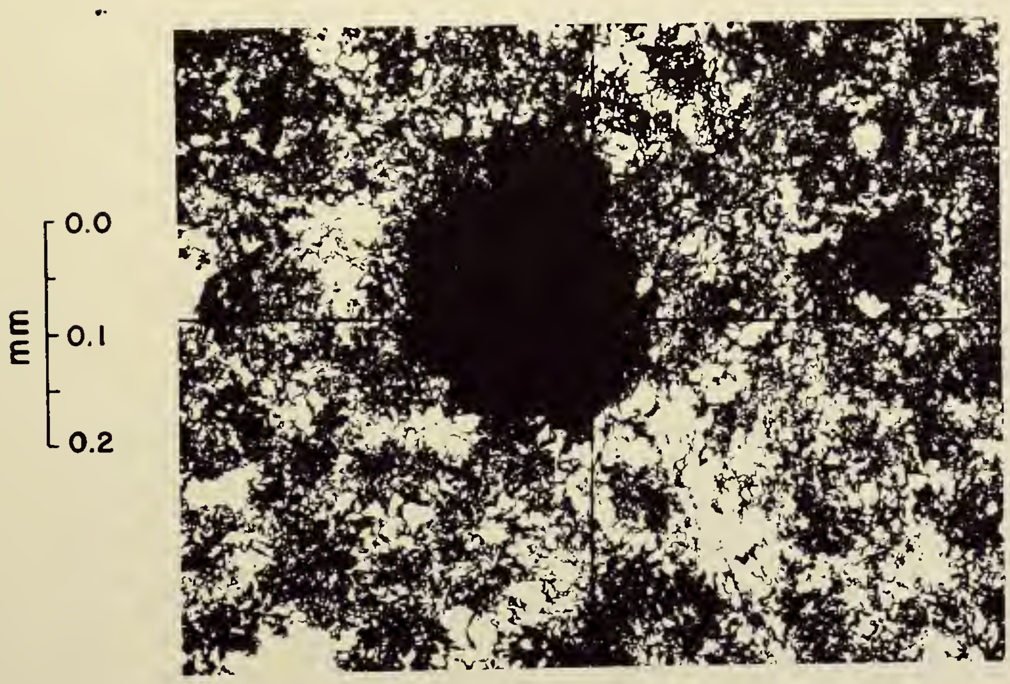


FIGURE 14.

Figure 15

Shale 2063 in plain light.
Detrital quartz grains up to 0.04 in diameter in fine-grained matrix consisting of clay minerals, limonite, and organic material.

Figure 16

Shale 2075 in plain light.
Opaque organic material in fine-grained matrix of clay minerals and limonite.
Light-colored grains averaging 0.01-0.02 mm. in diameter are detrital quartz.

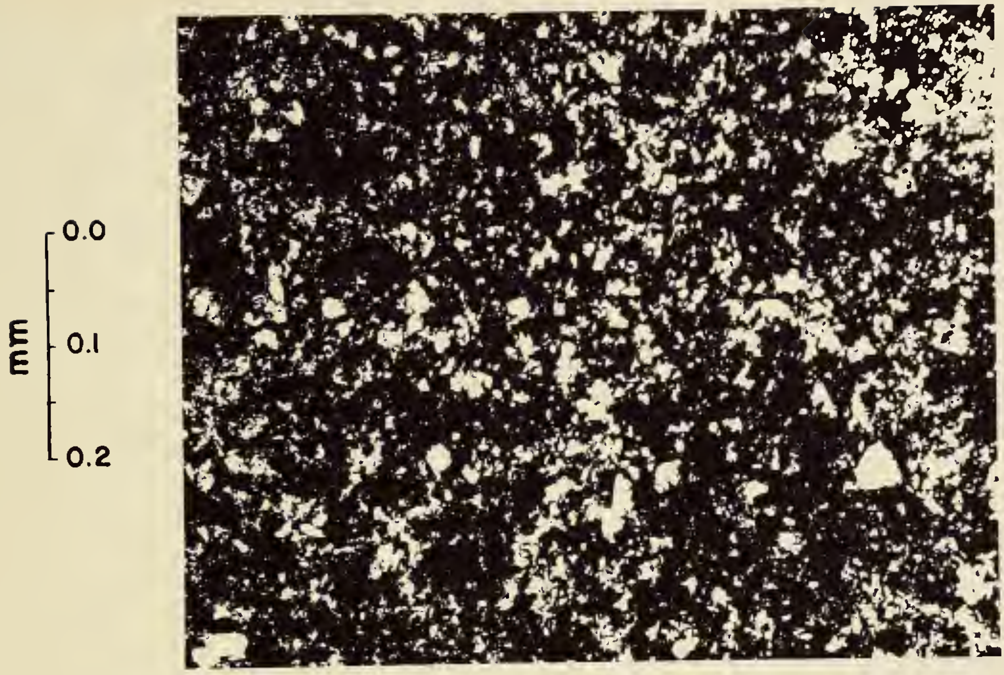


FIGURE 15.

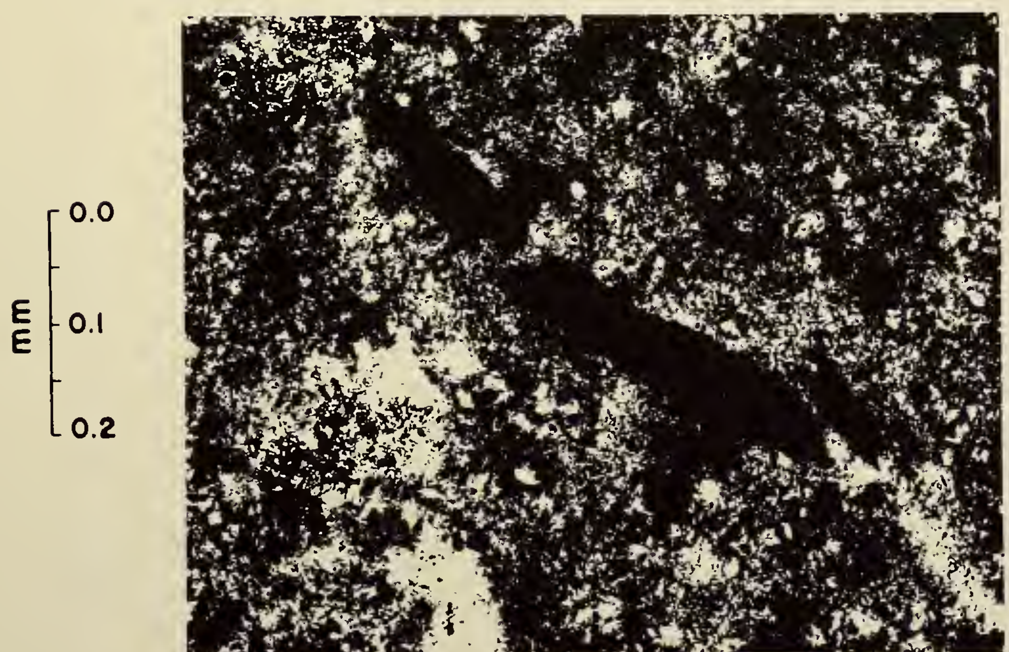


FIGURE 16.

Figure 17
and
Figure 18

Shale 2068 between crossed nicols.
These photomicrographs were taken to illustrate the preferred orientation of clay particles in shales.

Figure 17 shows the thin section at maximum illumination.

Figure 18, in which the microscope stage was rotated 45° from the position in Figure 17, shows the thin section in extinction.

The light-colored grains which are illuminated in both photographs are detrital quartz. The matrix consists primarily of clay minerals and limonite.

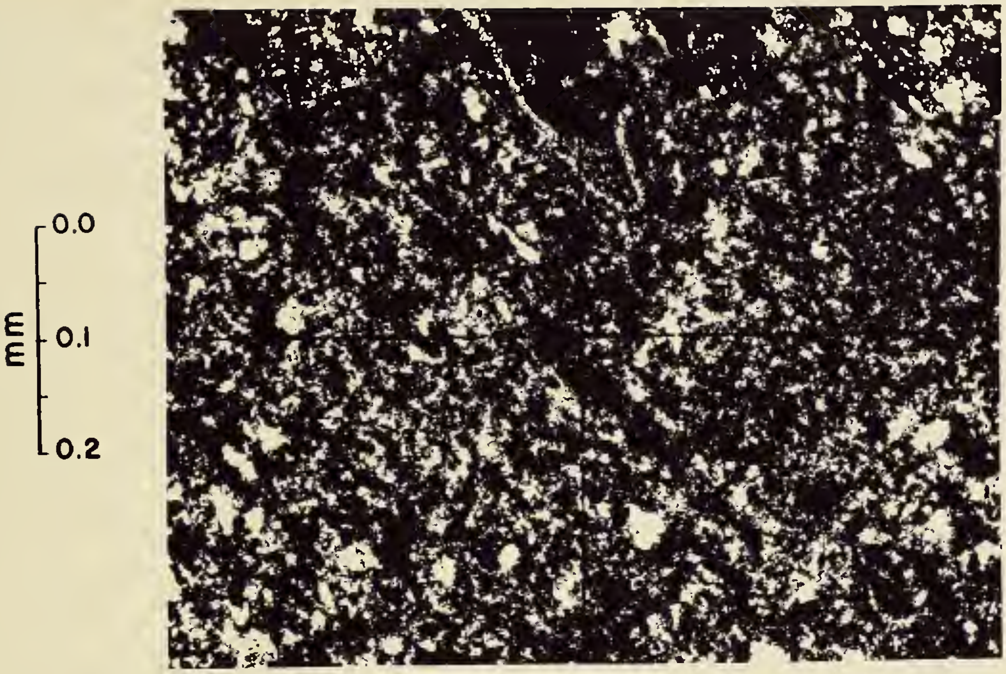


FIGURE 17.

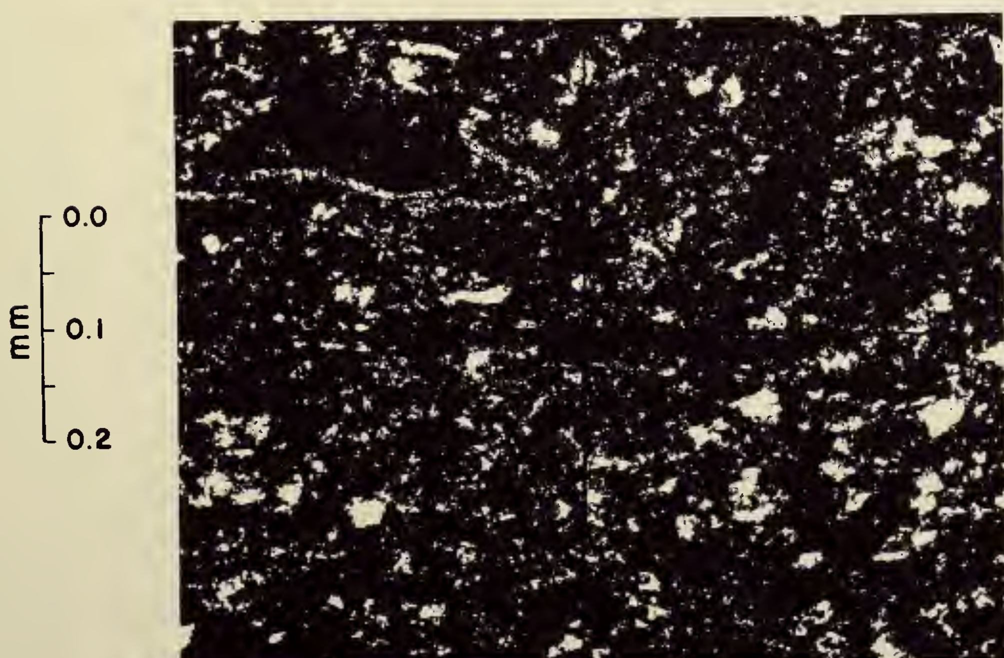


FIGURE 18.

RESULTS OF FREEZE-THAW TESTS

Durability Factors for Concrete Specimens

The results of freeze-thaw testing of the concrete beams are best presented in the form of the durability factors previously described. Table 15 presents durability factors for chert beams subjected to freeze-thaw testing. Durability factors for beams containing shale aggregates are shown in Table 16.

During the testing program, it was found that some of the crushed limestone used in the shale beams was of inferior quality and resulted in failure of certain beams. Beams which were suspected of having failed because of the limestone and not the shale were carefully broken by hand along the deep-seated cracks that had formed during freeze-thaw testing. Any beams that were found to have cracked because of deterioration of the limestone were omitted from the test results. The positions of such beams in the original experiment design are indicated in Table 16 by asterisks, and the omitted data are presented in Table 24 of Appendix B.

Plots of relative dynamic modulus of elasticity versus cycles of freezing and thawing for the complete freeze-thaw experiment on the chert beams are presented graphically in Figures 21 to 26 of Appendix B. Each of the curves represents the average relative dynamic modulus for the two beams of a particular cell of the experiment design. Figures

			(I	2077 (Ohio River)		
			Sp Gra	Specific Gravity Range		
			2.55 Plus	2.55 Plus	2.45-2.55	2.45 Minus
Per Cent Chert in Coarse Aggregate	2%		97.5	98.2	98.2	94.4
			98.3	97.8	96.5	95.5
		Avg.	97.9	98.0	97.4	95.0
	4%		99.8	97.2	97.3	90.3
			96.6	97.0	97.4	96.5
		Avg.	98.2	97.1	97.4	93.4
	6%					87.8
						68.9
		Avg.				78.4
	10%		98.2	97.9	96.3	30.0
			98.2	97.2	92.9	26.4
		Avg.	98.2	97.6	94.6	28.2

Table 15

Summary of Individual Durability Factors for Freeze-Thaw Testing Program of Concrete Beams Containing Small Percentages of Chert Coarse Aggregate

Per Cent Chert in Coarse Aggregate		Chert Source																	
		2063 (Decatur)			2064 (Elkhart)			2066 (Seymour)			2067 (West Lafayette)			2072 (Goaport)			2077 (Ohio River)		
		Specific Gravity Range			Specific Gravity Range			Specific Gravity Range			Specific Gravity Range			Specific Gravity Range			Specific Gravity Range		
		2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus
2%		97.5	98.2	94.1	98.2	98.2	96.4	98.8	97.3	89.1	97.9	96.9	96.1	98.2	97.0	93.8	98.2	98.2	94.4
		98.3	99.7	92.0	98.1	97.4	93.6	97.9	97.3	96.7	98.9	98.0	98.2	99.0	97.0	97.2	97.8	96.5	95.5
	Avg.	97.9	99.0	93.1	98.2	97.8	95.0	98.4	97.3	92.9	98.4	97.5	97.2	98.6	97.0	95.5	98.0	97.4	95.0
4%		99.8	98.3	88.9	98.2	97.3	95.5	98.0	96.5	95.5	97.7	96.4	95.5	99.0	94.6	96.5	97.2	97.3	90.3
		96.6	97.2	96.4	99.0	96.4	98.0	98.0	94.7	95.6	97.7	95.4	95.5	97.0	97.3	94.9	97.0	97.4	96.5
	Avg.	98.2	97.8	92.7	98.6	96.9	96.8	98.0	95.6	95.6	97.7	95.9	95.5	98.0	96.0	95.7	97.1	97.4	93.4
6%				96.3			94.7			88.7			84.5			92.1			87.8
				78.1			97.2			89.7			95.6			90.0			68.9
	Avg.			87.2			96.0			89.2			90.1			91.1			78.4
10%		98.2	71.0	24.4	99.1	82.5	11.2	97.0	93.5	29.6	99.1	83.2	57.3	98.9	92.0	64.7	97.9	96.3	30.0
		98.2	94.7	29.5	96.4	98.0	25.0	98.0	95.5	46.5	97.3	94.6	38.4	98.9	91.9	38.3	97.2	92.9	26.4
	Avg.	98.2	82.9	27.0	97.8	90.3	18.1	97.5	94.5	38.1	98.2	88.9	47.9	98.9	92.0	51.5	97.6	94.6	28.2

Table 16

Summary of Individual Durability Factors for Freeze-Thaw Testing Program of Concrete Beams Containing Small Percentages of Shale Coarse Aggregate

Per Cent Shale in Coarse Aggregate		Shale Source: 2063 (Decatur)	2066 (Seymour)	2068 (near LaPorte)	2075 (Indianapolis)	2076 (South Bend)
2%		100.0 98.8 92.0	97.8 96.0 92.8 94.5 95.3 *	99.5 85.5 94.0	96.5 * 88.5 97.2 97.2 98.2	96.0 96.5 96.0 95.4 95.8 96.3
2%	Avg.	97.0	95.3	93.0	95.5	96.0
4%		96.3 85.3 97.1	96.5 * 90.8 80.5 * 87.2	94.3 * * 97.1 96.5 95.3	96.4 96.4 97.2	96.3 94.0 97.2 93.0 96.0 99.5
4%	Avg.	92.9	88.8	95.8	96.7	96.0
6%		99.0 98.2 96.0 99.0 98.7 100.0	97.4 92.5 98.2 96.3 87.6 96.5	* 98.4 * 87.9 85.1 *	98.5 87.0 96.5	99.8 89.0 100.0 97.0 89.0 88.4
6%	Avg.	98.5	94.8	90.5	94.0	93.9
10%		95.5 86.5 95.0	87.0 96.5 * 97.0 96.0 96.2	* 83.0 * 95.3 93.5 91.6	* 98.0 96.2 96.4 98.2 96.4	90.8 95.4 95.5
10%	Avg.	92.3	94.5	90.9	97.0	93.9

* Deleted values are presented in Table 24 of Appendix B.

27 to 28 of Appendix B show comparable information for shale beams. Only those beams whose durability factors were included in Table 16 were used to obtain the curves shown here. Each curve represents the average relative dynamic modulus of elasticity of beams within a particular cell of the shale experiment design.

Surface Deterioration of Concrete Specimens

Surface deterioration factors were determined, as outlined previously, for each of the beams subjected to freeze-thaw testing. A summary of these factors for individual beams containing chert is presented in Table 17. The beams included in this tabulation are the ones whose durability factors are listed in Table 15.

The surface deterioration factors for the shale beams are shown in Table 18. These factors are for the same beams whose durability factors are presented in Table 16. As was the case for Table 16, the data for those beams that failed because of the presence of inferior limestone aggregates are not included in this table. The positions of these data in the experiment design are indicated by asterisks in Table 18, and the data are presented in Table 25 of Appendix B.

Comparison of Air Void Parameters in Hardened Concrete Mixed by Hand and by Machine

It was noted during the course of study that several beams cast from concrete mixed by hand produced lower durability factors than beams of identical composition for which the concrete was mixed in a machine mixer. It was felt that this presented an opportunity to study the air-

Per Cent Chert in Coarse Aggregate			(I Sp Grav	2066 (Ohio River) Specific Gravity Range		
			2.55 Plus	2.55 Plus	2.45-2.55	2.45 Minus
	2%		0.0	0.0	0.0	0.0
			0.0	0.0	0.0	0.0
		Avg.	0.0	0.0	0.0	0.0
	4%		0.0	0.0	0.0	1.7
			0.0	0.0	0.0	2.0
		Avg.	0.0	0.0	0.0	1.9
	6%					0.3
						0.0
		Avg.				0.2
	10%		0.0	0.0	0.0	1.5*
			0.0	0.0	0.0	0.0*
		Avg.	0.0	0.0	0.0	0.8

* Removed from

Table 17

Summary of Surface Deterioration Factors for Freeze-Thaw Testing Program
of Concrete Beams Containing Small Percentages of Chert Coarse Aggregate

Per Cent Chert in Coarse Aggregate		Chert Source																	
		2063 (Decatur)			2064 (Elkhart)			2066 (Seymour)			2067 (West Lafayette)			2072 (Goeport)			2066 (Ohio River)		
		Specific Gravity Range			Specific Gravity Range			Specific Gravity Range			Specific Gravity Range			Specific Gravity Range			Specific Gravity Range		
		2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus	2.55 Plus	2.45-2.55	2.45 Minus
2%		0.0	0.0	0.6	0.0	0.6	0.0	0.0	0.0	3.0	0.0	0.0	1.0	0.0	1.0	0.0	0.0	0.0	0.0
		0.0	0.0	0.0	0.0	0.0	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Avg.	0.0	0.0	0.3	0.0	0.3	0.2	0.0	0.0	1.5	0.0	0.0	0.5	0.0	0.5	0.0	0.0	0.0	0.0
4%		0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.6	0.0	0.0	0.0	1.0	0.0	0.0	0.0	0.0	0.0	1.7
		0.0	0.0	1.3	0.0	0.3	1.0	0.0	0.0	1.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	2.0
	Avg.	0.0	0.0	0.7	0.0	0.2	0.5	0.0	0.3	0.6	0.0	0.0	0.5	0.0	0.0	0.0	0.0	0.0	1.9
6%				1.0			2.6			1.7			0.3			1.7			0.3
				0.5			4.3			1.3			0.0			0.0			0.0
	Avg.			0.8			3.5			1.5			0.2			0.9			0.2
10%		0.0	0.0	0.0*	0.0	0.0	0.0*	0.0	0.0	2.5*	0.0	0.0	0.3	0.0	0.0	3.0	0.0	0.0	1.5*
		0.0	0.6	0.3*	0.0	0.0	0.0*	0.0	0.0	2.5*	0.0	0.0	0.0*	0.0	0.3	1.7*	0.0	0.0	0.0*
	Avg.	0.0	0.3	0.2	0.0	0.0	0.0	0.0	0.0	2.5	0.0	0.0	0.2	0.0	0.2	2.4	0.0	0.0	0.8

* Removed from freeze-thaw test at less than 300 cycles

Table 18

Summary of Surface Deterioration Factors for Freeze-Thaw Testing Program of Concrete Beams Containing Small Percentages of Shale Coarse Aggregate

Per Cent Shale in Coarse Aggregate		Shale Source: 2063 (Decatur)	2066 (Seymour)	2068 (near LaPorte)	2075 (Indianapolis)	2076 (South Bend)
2%		0.0	0.0	2.0	0.0	0.0
		0.0	0.0	2.0	*	0.0
		0.0	0.0	2.0	0.0	0.0
			0.0		0.0	0.0
			0.0		0.0	0.0
			*		0.0	0.0
	Avg.	0.0	0.0	2.0	0.0	0.0
4%		0.0	0.0	0.0	0.0	0.0
		0.0	*	*	0.0	0.0
		0.0	0.0	*	0.0	0.0
			0.0	6.0		0.0
			*	0.3		0.0
			0.0	1.7		0.0
	Avg.	0.0	0.0	2.0	0.0	0.0
6%		0.0	0.0	*	0.0	1.0
		0.0	0.5	4.2	0.0	0.0
		0.0	0.3	*	1.0	1.0
		2.0	2.0	0.3		0.0
		0.0	0.0	2.5		0.0
		2.0	0.0	*		1.0
	Avg.	0.7	0.5	2.3	0.3	0.5
10%		0.0	0.5	*	*	4.0
		0.0	1.0	1.2	0.0	3.3
		0.0	*	*	1.0	0.3
			0.0	5.9	0.0	
			0.0	3.0	1.0	
			0.0	8.5	0.0	
	Avg.	0.0	0.3	4.7	0.4	2.5

* Deleted values are presented in Table 25 of Appendix B.

void parameters of these two classes of concrete in an attempt to explain the observed differences.

By means of the linear traverse technique, the total percentage of entrained and entrapped air was determined for each beam. Using the values obtained for total percentage of air and voids per inch of traverse, it was possible to compute specific surface areas and bubble spacing factors for the beams using Powers' method (40). Table 19 presents the results of this air void study for beams containing shale. Figure 19 presents the same data graphically. A sample set of calculations for the specific surface area and the void spacing factor are included in Appendix C.

Table 19

Results of Air Void Studies of Certain Concrete
Beams by Means of the Linear Traverse Technique

Beam Number and Description	Durability Factor	% Air	Voids per Inch	Calculated Specific Surface Area of Voids (sq.in./cu.in.)	Calculated Void Spacing Factor (Inches)
S6-3 (4% Shale #2063, machine mixed)	97.1	3.3	4.2	506	.0080
S6-6 (4% Shale #2063, hand mixed)	21.1	4.1	2.9	287	.0131
S8-6 (10% Shale #2063, machine mixed)	95.0	3.8	4.8	512	.0075
S8-1 (10% Shale #2063, hand mixed)	6.0	3.8	2.0	208	.0185
S9-5 (10% Shale #2068, machine mixed)	83.0	2.7	3.5	523	.0088
S9-1 (10% Shale #2068, hand mixed)	8.3	3.3	2.7	324	.0126
S20-2 (10% Shale #2066, machine mixed)	96.5	3.5	3.8	441	.0091
S20-5 (10% Shale #2066, hand mixed)	14.2	3.0	2.0	269	.0165
S21-2 (6% Shale #2075, machine mixed	72.4	3.0	3.8	512	.0083
S21-6 (6% Shale #2075, hand mixed	10.6	3.1	2.1	273	.0154
S22-1 (10% Shale #2076, machine mixed)	90.8	2.8	3.2	451	.0098
S22-4 (10% Shale #2076, hand mixed)	30.1	2.7	2.2	326	.0139
S23-3 (10% Shale #2075, machine mixed)	96.2	3.1	5.0	637	.0066
S23-4 (10% Shale #2075, hand mixed)	5.3	3.8	3.0	316	.0121

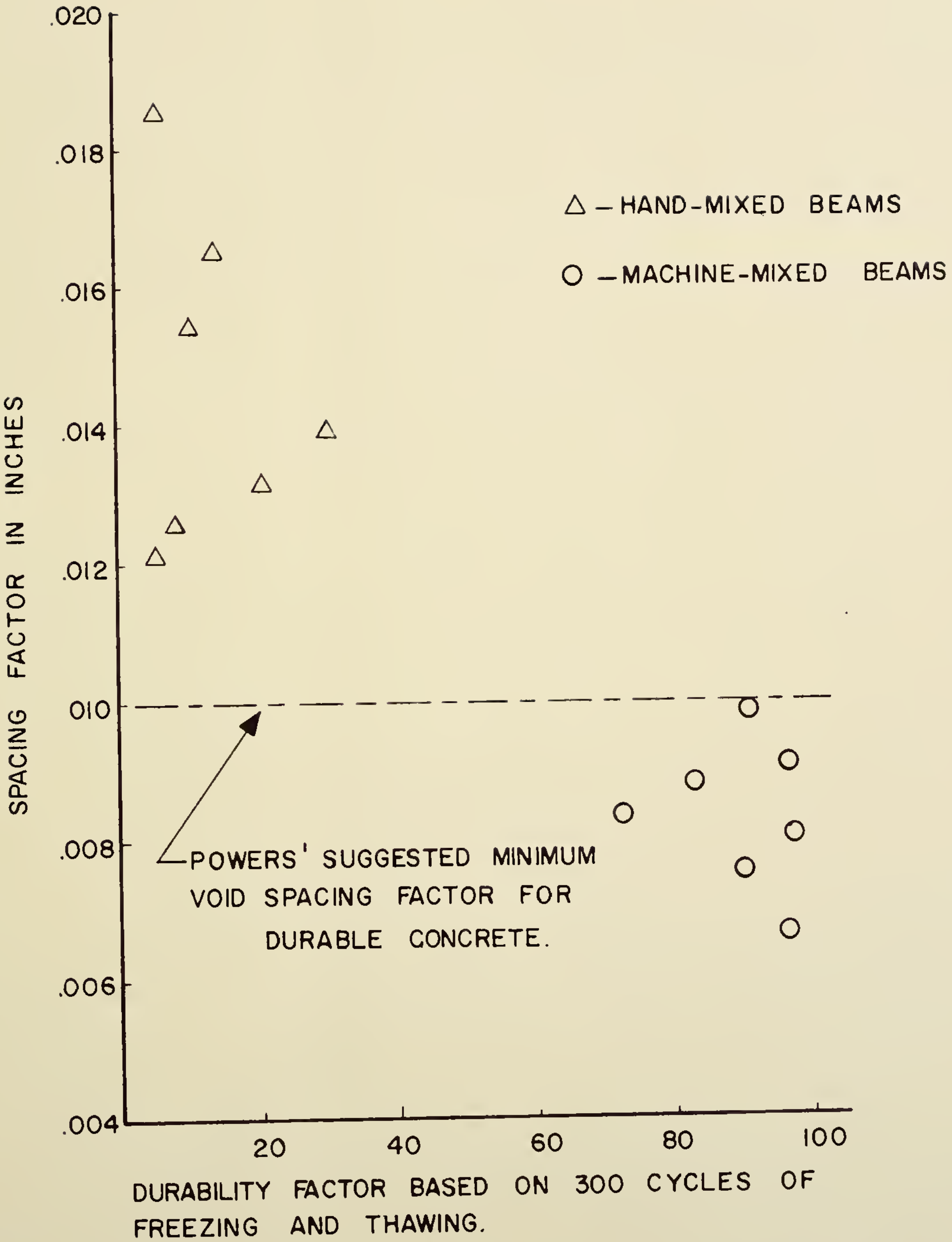


FIGURE 19. RELATIONSHIP OF DURABILITY FACTORS TO VOID SPACING FACTORS FOR AIR-ENTRAINED CONCRETE

STATISTICAL ANALYSIS OF RESULTS OF FREEZE-THAW TESTS

In this section statistical methods are used in analyzing differences in the durability factors of concrete specimens containing different percentages of cherts or shales from different sources, and in the case of the cherts, from different specific gravity levels. An attempt was made to determine the significance of the effect of each of the variables on the freeze-thaw durability of concrete. Such a study of the significance of variables on test results is commonly performed by means of a method known as the "analysis of variance" (3), which is a statistical procedure used for testing for significant differences among the means of the data.

The mechanics of the analysis of variance as applied to laboratory freeze-thaw test data for concrete are described in detail by Irick and Blackburn (18). Briefly, the analysis of variance is a method for determining whether observed differences in a set of data can be attributed to experimental factors or if the differences are due to uncontrolled variability in the data. The decisions concerning the causes of the observed differences are based on known probabilities. The experimental design for freeze-thaw studies of the chert beams was organized in such a way that the analysis of variance would be applicable to durability factors obtained in the tests. However, as is often the case in freeze-thaw testing, the variances for durability factors within the cells proved to be strongly non-

homogeneous when checked by means of Bartlett's test (3), thus eliminating the possibility of using the analysis of variance technique on the complete experimental design. However, Scheffe' (50) has indicated recently that the use of the analysis of variance on data whose variances are not homogeneous, but are not strongly non-homogeneous will result in a Type I error too small to affect the validity of the conclusions. Therefore, since most of the non-homogeneity of variances occurred in the six and ten per cent levels of this design, it was decided to run an analysis of variance limited to the two and four per cent levels crossed with sources and bulk specific gravity ranges in a three-way fixed crossed-classification. It was assumed that although the variances for the data at the two and four per cent levels are not homogeneous, they are close enough to homogeneity to permit use of the analysis of variance with little possibility of arriving at an incorrect conclusion due to Type I error.

The analysis of variance table for the chert beams is presented in Table 20. In addition to the information commonly included in an analysis of variance table, this presentation includes a summary of the hypotheses tested and the decisions reached on these hypotheses. It can be seen from this table that different sources of chert, as well as different percentages used in the blends (up to four per cent), and any interactions had no significant effect on the durability of of the concrete to laboratory freezing and thawing. The only variable causing significant differences in durability factors was

Table 20

ANOVA Table Based on Durability Factors for Concrete Beams Containing Two and Four Per Cent Chert

Source of Variation	Degrees of Freedom	Sum of Squares	Mean of Squares	$F_{obs.}$	$F_{\gamma_1, \gamma_2, \alpha}$	Decision
Source of Aggregate	5	8.61	1.72	0.54	$F_{5,36,0.05} = 2.48$	Accept H_{o_1}
Per Cent Blend	1	2.92	2.92	0.92	$F_{1,36,0.05} = 4.12$	Accept H_{o_2}
Source vs. Per Cent	5	6.05	1.21	0.38	$F_{5,36,0.05} = 2.48$	Accept H_{o_3}
Specific Gravity Range	2	132.69	66.35	20.8	$F_{2,36,0.05} = 3.26$	Reject H_{o_4}
Source vs. Gravity	10	38.31	3.83	1.20	$F_{10,36,0.05} = 2.12$	Accept H_{o_5}
Per Cent vs. Gravity	2	4.72	2.36	0.74	$F_{2,36,0.05} = 3.26$	Accept H_{o_6}
Source vs. Per Cent vs. Gravity	10	12.53	1.25	0.39	$F_{10,36,0.05} = 2.12$	Accept H_{o_7}
Error	36	114.72	3.19			
Total	71	320.55				

Hypotheses:
H_{o_1} = no difference between sources of chert
H_{o_2} = no difference between percentages in blends
H_{o_3} = no interaction between sources and percentages
H_{o_4} = no difference between saturated, surface-dry bulk specific gravity ranges
H_{o_5} = no interaction between sources and gravity ranges
H_{o_6} = no interaction between percentages and gravity ranges
H_{o_7} = no interaction among sources, percentages, and gravity ranges.

bulk specific gravity. This is demonstrated in the following table of mean durability factors obtained by averaging the values for all six chert sources:

Bulk Specific Gravity Range (Saturated Surface-Dry Basis)	Two Per Cent Chert	Four Per Cent Chert
2.55 Plus	98.2	97.9
2.45-2.55	97.6	96.6
2.45 Minus	94.8	94.9

However, it should be noted that while these differences in durability factors may be statistically significant, the durability factors for the two and four per cent blends of the 2.45 minus chert are above 90 which is high enough to denote sound concrete after 300 cycles of freezing and thawing. Thus while the analysis of variance has been able to indicate significant differences between specific gravity ranges at the two and four per cent levels, this technique does not differentiate between sound and unsound concrete at these levels.

Study of the complete outline of durability factors indicates that the analysis of variance does not provide all the information to be obtained from these data. It is obvious that the lowest durability factors occur for those beams containing six and ten per cent of 2.45 minus chert, and the analysis of variance could not be used to bring out differences between these and other cells because of the strong non-homogeneity of variances in the six and ten per cent levels. In order to test differences between means of the six and ten per cent

levels and the two and four per cent levels, the Fisher-Behrens test (3) was utilized. This method is intended for determining significant differences between means when the variances are equal and unknown.

As shown in Tables 26 to 29 of Appendix B, the Fisher-Behrens tests were conducted against a critical $t_{2\alpha}$ (one-sided test) for a level of significance $\alpha = 0.01$. The comparisons made and conclusions arrived at are as follows:

(a) At the 2.55 plus specific gravity level the mean durabilities for the ten per cent blends were not significantly lower than those of the two and four per cent blends.

(b) At the 2.45-2.55 specific gravity level the mean durabilities for the ten per cent blends were not significantly lower than the means for the two and four per cent blends.

(c) At the 2.45 minus specific gravity level the means for the ten per cent blends were significantly lower than the means for the two, four, and six per cent blends.

(d) At the 2.45 minus specific gravity level, the means for the six per cent blends were not significantly lower than the means for the two and four per cent blends.

Since all the durability factors for the shale beams were high enough (Table 16) to indicate sound concrete (the lowest cell average was 89) statistical analysis of these data was not warranted. By the same token, the significance of the data on surface deterioration

(Tables 17 and 18) was self-evident and no further analysis was needed.

The results of statistical analyses of the durability factors for the chert beams may be summarized as follows:

1. Sources of chert had no effect on durability factors.

2. Percentages of chert up to four per cent had no effect on durability factors at any specific gravity level. In addition:

    a) At the 2.55 plus and 2.45-2.55 specific gravity levels percentages of chert up to ten per cent had no effect on durability factors.

    b) At the 2.45 minus specific gravity level, percentages up to six per cent had no effect on durability factors, but ten per cent chert resulted in significantly lower durability factors.

3. Interactions among sources, percentages, and specific gravity levels were not significant.

DISCUSSION OF RESULTS OF FREEZE-THAW TESTS OF CHERTS AND SHALES IN CONCRETE

The primary purpose of this study was to determine the effect of cherts and shales from different geographical areas on the freeze-thaw durability of concrete containing small percentages of these materials. In order to compare the freeze-thaw resistance of cherts and shales from different areas, two criteria were used: (a) resistance to deep-seated deterioration as denoted by durability factors, and (b) resistance to surface deterioration (popout and pitting damage).

Durability Factors

Statistical analysis of durability factors obtained from freeze-thaw studies of the chert beams (Table 15) indicated that the sources of the chert had no effect on resistance of concrete to deep-seated freeze-thaw deterioration. Even though the cherts were from different sources throughout Indiana, they resulted in nearly equal degrees of freeze-thaw deterioration when used in concrete in equal amounts having the same specific gravity ranges.

A definite difference in durability factors was found, however, for beams containing cherts of different specific gravity ranges. It was found that beams containing chert from the 2.45 minus specific gravity group had significantly lower durability than those containing

chert from the 2.55 plus and 2.45-2.55 specific gravity ranges. This is in accord with the work of Sweet and Woods (60) which indicated low-specific gravity chert to be the most susceptible to freeze-thaw deterioration.

The percentage of chert used also had a significant effect on the durability factors, but only at the 2.45 minus specific gravity level. At the 2.55 plus and 2.45-2.55 specific gravity levels, there was no significant difference among the durability factors of the beams containing different percentages of chert. Even ten per cent of chert from these specific gravity groups caused no deep-seated failure of beams in which it was included.

In the 2.45 minus specific gravity level the percentage of chert had a strong effect on durability of the concrete. Without exception, beams containing ten per cent of 2.45 minus chert suffered severe deep-seated deterioration. Every beam was intersected by at least one deep-seated crack caused by failure of the chert. The lowest durability factors recorded in the freeze-thaw testing occurred for this combination.

At the six per cent level of the 2.45 minus specific gravity group, no deep-seated cracks occurred and the durability factors were found to be not significantly lower than those of the two and four per cent levels of this gravity range. However, more variability in the data occurred at this level than at the two and four per cent levels, i.e., individual beams containing six per cent of 2.45 minus chert had durability factors as low as 68.9 and 78.1 while others were as high as 97.2. The few low durability factors at the six per

cent level, while not nearly as low as those at the ten per cent level and not low enough to cause significant differences in the cell means, were low enough to suggest that some deterioration can occur in concrete containing as little as six per cent chert with a bulk specific gravity of less than 2.45.

Durability factors for beams containing two and four per cent of 2.45 minus chert were shown by analysis of variance to be significantly lower than durability factors for beams containing the same percentages of chert from the two heavier specific gravity ranges. However, all the durability factors for these beams are high enough to indicate very little deep-seated deterioration (Table 15). For example, the lowest durability factor computed for the four per cent level of the 2.45 minus specific gravity range was 88.9 and the lowest cell mean was 92.7. These values are high enough to be considered indicative of sound concrete.

In summary, it appears that concrete containing up to four per cent chert with a bulk specific gravity (saturated surface-dry basis) of 2.45 or less, and at least ten per cent chert with a specific gravity greater than 2.45, can successfully withstand laboratory freeze-thaw exposure without undergoing deep-seated deterioration.

The effect of size of the individual chert particles on their freeze-thaw durability in concrete is of interest. Previous studies of deleterious substances have indicated an apparent relationship between size of unconfined particle and lack of freeze-thaw durability. Wray and Lichtefeld (73) found in their study of unconfined Missouri cherts that saturated 1 to $1\frac{1}{2}$-inch particles had less resistance to

freezing-and-thawing failure than saturated 3/4 to 1-inch particles. Thomas (62) saturated prisms of different sizes from the same rock and found that damage was greater the larger the specimen.

The effect of size on the durability of particles of deleterious materials in concrete is not so clear, however. Sweet and Woods (60) embedded saturated chert pieces of three sizes, 3/4 to 1 inch, 1/2 to 3/4 inch, and 3/8 to 1/2 inch, in 1-inch mortar cubes and subjected these cubes to up to 309 cycles of freezing and thawing. They found that the cubes failed at an earlier cycle for the 3/4 to 1-inch pieces than for the 1/2 to 3/4-inch pieces, and that no failure occurred in the cubes containing the 3/8 to 1/2-inch pieces. Klieger (21), however, found no apparent relationship between size of unsound aggregate particles and durability as long as the air content of the mortar was held constant. R. Walker and McLaughlin (65) demonstrated that light-weight chert less than 3/8-inch in size would not cause deep-seated freeze-thaw deterioration in concrete, but their method of test did not distinguish between the degrees of resistance to freeze-thaw deterioration exhibited by different sizes of chert larger than 3/8-inch.

In this study no attempt was made to use different sizes of chert in different beams to determine the effect of size on durability. However, for those beams that had suffered deep-seated cracking as a result of freezing and thawing, a qualitative study was conducted to determine the sizes of the pieces of chert intersected by each crack with the hope of determining which size (3/8 to 1/2-, 1/2 to 3/4-, or 3/4 to 1-inch) had caused the most deep-seated deterioration. This

study showed that each of the cracks intersected a piece of 3/4 to 1-inch chert, and that each crack appeared to have been caused primarily by this large piece of chert. Most of the cracks also intersected one or more smaller pieces of chert which probably aided the 3/4 to 1-inch piece in causing the failure, but in each of these cases it appeared that the 3/4 to 1-inch piece had provided most of the disruptive force. In a few cases, cracks intersected only the 3/4 to 1-inch piece of chert, and no smaller pieces; and in no case was a crack caused by 1/2 to 3/4- or 3/8 to 1/2-inch pieces alone. This, of course, does not mean that 1/2 to 3/4- or 3/8 to 1/2-inch pieces could not cause deep-seated failure of concrete, but that they are not as harmful as the larger pieces. In summary, it appears that larger chert particles in concrete have less resistance to freeze-thaw deterioration than smaller ones.

Study of the durability factors for concrete beams containing shale (Table 16) indicates that no combination of sources and percentages (up to ten per cent) of shale resulted in deep-seated failure of the beams. Only a few beams were found to have durability factors below 90, and these few values appear to be well distributed throughout the data. Only one cell mean is below 90 and this is at the four per cent level, while the durability factors at the six and ten per cent levels for this same shale (2066) are well above 90. This indicates that the low mean for the four per cent level is probably due to random error.

Thus it appears from these data that in amounts of up to ten per cent, little difference in the resistance of concrete to deep-

seated deterioration was caused by the different shales even though they were from widely separated areas throughout the state and had significantly different basic properties. This is in accord with the findings of Lang (25) who noted that for pavement concrete containing small percentages of shale, the only harmful effect of the shale due to freezing and thawing consisted of surface deterioration of the concrete.

A comparison of durability factor data for the cherts and shales shows that the only deep-seated deterioration caused by either of these materials was due to chert with a bulk specific gravity (saturated surface-dry basis) of less than 2.45. Since some of the shale samples contained a considerable quantity of material which is of low bulk specific gravity even for shale (2.15 minus or 2.25 minus), and since none of these shales produced any deep-seated failure, specific gravity apparently does not have the same relationship to resistance to deep-seated failure for shales as it does for cherts.

Surface Deterioration

Although freeze-thaw testing of concrete specimens is primarily intended to cause deep-seated failure and subsequent loss of strength of concrete specimens containing unsound aggregates, surface deterioration of the concrete, which can be equally as important as deep-seated failure, often occurs in these tests. A part of this study was to determine how surface deterioration is influenced by each of the variables introduced into the freeze-thaw study.

Surface deterioration factors for the chert beams are presented

in Table 17. These data indicate no significant difference in severity of popout damage among the six chert sources. The major differences in severity of surface deterioration apparently were caused by material from different bulk specific gravity ranges. For all six cherts a negligible amount of surface deterioration occurred in beams containing material from the 2.55 plus and 2.45-2.55 specific gravity ranges. Material from the 2.45 minus gravity range, however, resulted in a significant amount of popout damage in beams made from each of the six cherts. In general, the six to ten per cent levels within the 2.45 minus specific gravity range had higher surface deterioration factors than the two and four per cent levels, but this was not true for all six cherts. For example, in the 2.45 minus specific gravity range for chert 2077, the four per cent level had an average surface deterioration factor of 1.9, while the six per cent level had an average factor of only 0.2. This seeming anomaly is probably due more to random positioning of the pieces of chert than to any error in procedure or real differences in the material.

It also should be noted that, in most cases, larger factors were obtained for beams in the six per cent level of the 2.45 minus specific gravity range than in the ten per cent level. This anomaly was primarily due to failure of most of the ten per cent beams to complete a full 300 cycles of freezing and thawing while all the six per cent beams lasted the full 300 cycles. Thus the ten per cent specimens were not exposed to as many cycles of freezing and thawing as those containing six per cent chert.

In summary, surface deterioration in the beams containing

chert paralleled the deep-seated failure of these beams. In both cases the different sources had little, if any, effect. For all sources failure occurred primarily in the six and ten per cent levels of the 2.45 minus specific gravity range.

Surface deterioration factors for the shale beams are presented in Table 18. It is evident from these data that shale 2068 caused considerably more popout damage than any of the other shales. Beams containing shale 2068 had higher surface deterioration factors than beams containing the other shales at every percentage level, and shale 2068 was the only shale to cause even a single popout in beams containing two to four per cent of this material. Apparently the relatively severe surface damage caused by shale 2068 was due to the greater porosity and absorptivity of this shale as compared to the others (Tables 10 and 13).

Only a small difference in performance relative to popout damage could be detected among the other four shales. No surface deterioration occurred for any of these shales when used in amounts up to and including four per cent. At the six to ten per cent levels, shales 2066 and 2076 caused a little more popout damage than shales 2063 and 2075, but the difference is slight.

Comparison of surface deterioration factors for the cherts and shales indicates that, except for sample 2068, the shales caused about the same amount of surface deterioration as cherts of the 2.45-2.55 specific gravity range. In general, the shales caused greater popout damage than cherts of the 2.55 plus specific gravity range and lesser damage than cherts of the 2.45 minus gravity range. Shale 2068, however, resulted in more severe surface deterioration than any of

the cherts of any specific gravity range.

Air Voids in Concrete by Linear Traverse Technique

The results of this study, as shown in Table 19 and Figure 19, confirm the theoretical air voids concepts developed by Powers (40). Powers theorized that the increase in durability afforded concrete by means of air entrainment is largely a function of the spacing of the air voids in the concrete. He suggested that a concrete containing air voids with high specific surface area, and thus with a low void spacing factor, would receive more protection from the air voids than one containing air bubbles having a low specific surface area and a high spacing factor. He considered a void spacing of about 0.01 inches to be critical; those concretes with spacing factors lower than 0.01 inch were thought to be well protected from freezing-and-thawing deterioration; those with spacing factors greater than 0.01 inches were thought to be poorly protected.

This theory is supported by the results of the linear traverse studies. For the 14 beams studied, it was found that those having high durability factors (83 and above) all had spacing factors below 0.01 and those with low durability factors (21 and below) had spacing factors above 0.01. Also, the specific surface areas of the beams with high durability factors were all higher than those with low durability factors.

INFLUENCE OF BASIC PROPERTIES ON FREEZE-THAW DURABILITY

The primary objective of this portion of the study was to learn more about the basic properties of cherts and shales in Indiana's gravel aggregates and to determine how these properties affect the freeze-thaw durability of these materials. This section will relate the results of the studies of properties of the cherts and shales to the durability of these materials as determined by freeze-thaw testing. The properties to be discussed are porosity, absorption, mineralogy, and texture and microstructure.

Porosity

Much work relating porosity and durability of crushed limestone aggregates has been accomplished by Sweet (59) and Fears (13). Early studies by Cantrill and Campbell (9), Wuerpel and Rexford (74), and Sweet and Woods (60) correlated porosity and durability for cherts. However, little correlation of this type has been attempted for shales, and the chert studies mentioned were made before air-entrained concrete had come into use and before freeze-thaw testing had reached its present state of development. In addition, little was done in a quantitative sense in these previous studies. For example, it was determined in a qualitative way that lightweight chert causes deterioration when used in concrete exposed to freezing-and-thawing,

but nothing has been done to determine the quantity of this material required to cause deterioration.

Total Porosity

The total porosity of chert is inversely related to its bulk specific gravity (a more easily measured characteristic than porosity) for materials of the same true specific gravity, and as such is generally reflected in specifications for chert in concrete aggregate. It has been found that the most porous cherts (those with the lowest bulk specific gravities),cause the most severe freeze-thaw deterioration.

In this study, each chert sample was divided into bulk specific gravity ranges before being used in the concrete. The freeze-thaw studies of concrete beams containing chert from each of these specific gravity groups showed that deep-seated and surface deterioration took place only in beams containing six to ten per cent of material from the 2.45 minus specific gravity group. For the samples tested, the porosity of chert in this specific gravity group was nearly 13 per cent. Chert from the 2.45-2.55 and 2.55 plus specific gravity groups, which caused very little freeze-thaw deterioration, had porosities of only about seven and three per cent respectively. This relationship definitely supports the ideas of Wuerpel and Rexford (74) and Sweet and Woods (60) that cherts with high porosity are more susceptible to freeze-thaw deterioration than those with low porosity, and further demonstrates that this concept holds for air-entrained concrete as well as concrete with no entrained air. It also suggests

that the 2.45 bulk specific gravity level (saturated surface-dry basis) suggested by Sweet and Woods as the critical level of separation between unsound chert and durable chert is realistic even for air-entrained concrete.

The lack of protection afforded porous aggregates such as these lightweight cherts by air-entrained cement paste has been explained in general terms by Powers (39). When saturated aggregate particles surrounded by air-entrained cement paste are subjected to freezing, the water in the paste is able to move to the "escape boundaries" provided by entrained bubbles in the paste, and no excess hydraulic pressures are able to develop. Thus the paste itself is protected from dilation[1]. However, saturated porous rock particles enclosed by the paste still perform as virtually enclosed containers and are only a little better off than if the paste were not air-entrained. Probably the paste bubbles near the contact between aggregate particle and paste do accept a small amount of the excess water produced by freezing the saturated aggregate, but for saturated aggregates of high porosity, the amount of excess water is too large to be taken on by the bubbles in the paste immediately adjacent to the aggregate. For this reason, protecting the paste by air entrainment, while possibly successful for aggregates of low porosity, fails to protect saturated highly porous aggregate particles. This concept helps to explain the findings of Axon, Willis, and Reagel (2) who noted that the entrainment of air resulted in a definite improvement in durability

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[1] Volume change produced by freezing water in concrete.

of concrete containing limestones with good service records, but only caused a slight increase in durability for concrete made with chert-rich aggregate with a fair service record, and affected no appreciable improvement in durability of concrete made with chert-rich aggregate with a poor service record.

As shown in Table 10, the porosities of the different shales varied widely. For example, shale 2068, which was the softest and weakest of the shales, was nearly twice as porous as any of the other shales, and over five times as porous as shale 2063, the least porous and the most-indurated of the samples. The widely varying porosities of the shales had no effect on the amount of deep-seated freeze-thaw deterioration caused by these materials, however. In amounts up to ten per cent, none of the shales caused any deep-seated damage to the concrete in which they were used. This lack of deep-seated deterioration of concrete containing shales of relatively high porosity was due to the inherent structural weakness of these materials. Since the shale is considerably weaker than the surrounding mortar, it will fail internally due to the pressures developed in freezing rather than disrupt the mortar. This was demonstrated by comparison between pieces of chert and shale that had failed in the freeze-thaw test and subsequently had been removed from the beams. The chert pieces, which had caused deep-seated deterioration, had broken into many pieces, but the individual pieces were still relatively hard and firm. The shales, on the other hand, had disintegrated into weak crumbly masses although they had caused no deep-seated deterioration. From all appearances these shales had failed internally before the pressures

due to freezing could develop enough to break the surrounding mortar.

Where an individual shale particle occurred close to the surface of a beam, the enclosing mortar layer was often not strong enough to resist the hydrostatic pressures developed by freezing the saturated particle. In this case the mortar was disrupted, resulting in surface deterioration in the form of a popout or pit. The relative porosities of the shales had a marked relationship to severity of surface deterioration. Shale 2068, the material having the highest porosity of the group (Table 10), caused considerably more popout damage than any of the other shales or any of the cherts. It appears that this larger amount of deterioration is related to the greater porosity of shale 2068, but, as will be brought out in a succeeding section, other factors such as the size of the pores and the amount of absorption of the shale probably have a greater effect on the durability of the aggregate than the total porosity.

It should be noted that among the other four shales the relationship between porosity and surface deterioration is not so clear. Shales 2076 and 2066 are considerably more porous and more absorptive than 2063 and 2075, yet caused only a little more popout damage than 2063 and 2075.

In summary of the relationship of total porosity to freeze-thaw deterioration of concrete containing cherts and shales, the following points should be brought out:

(a) Although other pore characteristics such as pore size and absorptivity may have a strong influence on the resistance of the cherts tested to both deep-seated and surface deterioration, there is a definite relationship between

total porosity and the freeze-thaw resistance of these materials. The more porous fractions from all six chert groups caused more freeze-thaw deterioration than the less porous material.

(b) Total porosity of shales was related to severity of surface deterioration of concrete in which the shales were used, but in spite of widely varying porosities, none of the shales resulted in deep-seated deterioration of concrete in which they were used in amounts up to ten per cent. Shale 2068, which was considerably more porous than the other shales, caused much more surface deterioration than the others but caused no deep-seated deterioration.

Size of Pores

Although recognizing a relationship between total porosity and freeze-thaw durability of aggregates, Sweet (59) and Fears (13) have contended that the durability of aggregates is dependent more on the size and continuity of aggregate pores than on total porosity. Lewis and Dolch (27) maintained that the harmful pore size is that large enough to permit water readily to enter much of the pore space but not large enough to permit easy drainage. Studies by Sweet (59), and Fears (13) have indicated that critical pore size for freezing-and-thawing durability of limestone aggregates is about 5 microns. Blanks (5) has shown that, under natural conditions of freezing and thawing, voids less than 5 microns in diameter, and particularly those less than 4 microns in diameter, will drain effectively only at hydrostatic pressures that exceed the tensile strengths of some rocks and concrete.

These previous investigations indicate the importance of microvoids (pores less than 5 microns in diameter) in the durability of aggregates. Therefore part of the present study was devoted to determining if this relationship between pore size and durability holds for Indiana cherts. For cherts 2067 and 2077, the percentage of total volume of aggregate occupied by voids greater than 5 microns in diameter was determined by a linear traverse method, and this percentage was subtracted from the total porosity to obtain the percentage of total aggregate volume occupied by microvoids. It was found that the volume of microvoids was somewhat less than expected. Sweet (59) had noted that in Indiana limestone aggregates the volume of microvoids, expressed as a ratio of the total volume, was less than 0.057 for aggregates with good field performance records and greater than 0.091 for aggregates with poor service records. If Sweet's criteria were to be applied to the chert fractions whose void ratios are presented in Table 11, it would seem that none of the material in these fractions would be susceptible to freeze-thaw deterioration since none of this material has microvoid ratios[1] as high as 0.091. The highest ratio, 0.064 for the 2.45 minus specific gravity group of chert 2067, is only slightly higher than the 0.057 ratio designated as the upper limit for aggregates with good service records. In spite of these relatively low microvoid ratios, the 2.45 minus specific gravity chert from sources 2067 and 2077 caused serious freeze-thaw deterioration in concrete in

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[1] As used here the term "microvoid ratio" refers to the ratio of volume of voids less than five microns in diameter to the bulk volume of the aggregate.

which it comprised ten per cent of the coarse aggregate.

It also should be noted that practically no freeze-thaw deterioration occurred in concrete containing chert from the 2.45-2.55 specific gravity range even though this material contained nearly as large a ratio of microvoids as did chert from the 2.45 minus specific gravity group. In spite of the lack of difference in volume of microvoids between the 2.45-2.55 and 2.45 minus specific gravity ranges, there is considerable difference in total porosity between these ranges. As shown in Table 21, the high total porosity of the 2.45 minus chert as compared to the 2.45-2.55 material is primarily due to the increase in voids larger than 5 microns in diameter in the 2.45 minus material. Table 21 shows that as the total porosity of the chert increases in going from material of high to low bulk specific gravity, the voids larger than 5 microns in diameter constitute an increasingly larger percentage of the total pore space, and conversely, the microvoids make up an increasingly smaller percentage of the pore space. For example, for chert 2067, the microvoids constitute 80 per cent of the total pore space in the 2.55 plus chert, 75 per cent in the 2.45-2.55 material, and only 50 per cent in the 2.45 minus range.

On the basis of this study, it appears the microvoids ratio is not as reliable an indicator of freeze-thaw durability of chert aggregate as Sweet (59) and Fears (13) found it to be for limestone aggregate. The results of this study indicate that some other pore characteristic or, more probably, a combination of characteristics (perhaps including volume of microvoids) is probably the main factor in determining chert durability.

Table 21

Relation of Pore Size to Degree of Saturation for Cherts 2067 and 2077

Source	Specific Gravity Range	Total Porosity (per cent)	Per Cent of Bulk Volume Consisting of Voids > 5 Microns in Diameter	Per Cent of Voids Volume Consisting of Voids > 5 Microns in Diameter	Per Cent of Bulk Volume Consisting of Voids < 5 Microns in Diameter	Per Cent of Voids Volume Consisting of Voids < 5 Microns in Diameter	Degree of Saturation (per cent)
2067	2.55 plus	3.0	0.6	20.0	2.4	80.0	82.3
	2.45-2.55	7.6	1.9	25.0	5.7	75.0	92.5
	2.45 minus	12.9	6.5	50.4	6.4	49.6	100.0
2077	2.55 plus	3.0	0.4	13.3	2.6	86.7	78.3
	2.45-2.55	6.4	2.3	35.9	4.1	64.1	87.7
	2.45 minus	12.8	7.9	61.7	4.9	38.3	90.5

As shown in Table 21, the degree of saturation of the cherts increases with increasing total porosity and decreasing bulk specific gravity. This increase is probably caused by the larger percentage of voids larger than 5 microns in diameter in the more porous material. Under conditions of vacuum saturation and frequently repeated immersion as used in these tests, a piece of aggregate containing numerous large voids as well as microvoids would probably reach a high degree of saturation more readily than a particle containing only microvoids, because of the greater ease of flow through the larger voids. In this way it is thought that 2.45 minus chert becomes more saturated than heavier less porous fractions, and that this high degree of saturation is a prime factor in the lack of durability of the 2.45 minus material.

Besides being a factor in the permeability of the chert, the size of the pores undoubtedly determines whether dilation occurs. Pores or bulges in pores that are large enough to act as escape boundaries for the water under hydrostatic pressure (39) will cause no dilation. Obviously some of the large pores in the 2.45 minus material are in this category. The critical size between pores which will cause dilation and those which will act as escape boundaries depends on the length and tortuosity of the pores. It is possible that dilation in lightweight cherts occurs entirely within voids less than 5 microns in diameter which are supplied with water by the larger voids, but it is quite probable that some of the voids larger than 5 microns are too small to serve as escape boundaries and thus contribute to dilation of the particle.

Of course there is always the possibility that freeze-thaw failure of concrete test beams in tests such as these is caused by individual aggregate pieces which vary considerably from the norms of

their groups in porosity characteristics. Thus it could be postulated that freeze-thaw failures involving cherts of the 2.45 minus specific gravity range are caused by a relatively few pieces having percentages of microvoids considerably greater than the average values for this group. In an attempt to either validate or disprove this postulation, vacuum-saturated absorption tests were conducted on fragments of several individual pieces of chert which were taken from deep-seated cracks in test beams, and which in every case had caused these cracks. The absorption tests were used because the small size of the fragments precluded the use of direct measurements of porosity. Comparison of the percentages of absorption obtained for these individual pieces of chert (Table 14) with the absorption values of the 2.45 minus specific gravity groups (Table 12) indicates that while the absorption values for the individual pieces are in most cases a little higher than the values for the groups, the difference is not great and the relationship does not hold for all six cherts. Therefore the results of this test apparently reject the theory that the deep-seated failures were due to extremely porous and highly absorptive individual pieces within the 2.45 minus specific gravity group. Instead pieces having only average absorptions for this gravity group are able to cause deep-seated failure.

In summary, the microvoids ratio does not provide a satisfactory indication of the freeze-thaw durability of chert. Instead chert durability is probably more closely related to the degree of saturation of these small voids (and larger voids that are too small to act as escape boundaries) which is strongly influenced by the presence

of voids greater than 5 microns in diameter. Based on this concept and the results of the freeze-thaw tests, it appears that total porosity, as reflected in bulk specific gravity, serves as a satisfactory criterion for predicting the freeze-thaw durability of chert.

Absorption

Lewis and Dolch (27) have stated that, "The lack of durability of an aggregate in freezing and thawing is primarily dependent upon its ability to become and stay highly saturated under the given conditions of moisture." Thus besides being porous, an aggregate must be absorptive in order to be susceptible to freeze-thaw deterioration. This section discusses the relationship between (a) vacuum-saturated absorption and (b) rate of absorption of cherts and shales, and the resistance of these materials to freeze-thaw deterioration.

Vacuum-Saturated Absorption

Comparison of the results of the vacuum-saturated absorption tests of the chert groups (Table 12) to the results of freeze-thaw tests of concrete containing the cherts indicates a direct relationship between percentage of absorption and lack of freeze-thaw durability for chert. Cherts of the 2.45 minus specific gravity ranges for all six sources had absorption percentages about twice as great as those for the 2.45-2.55 groups and five times as great as the 2.55 plus groups. The absorptions of these materials are directly related to their porosities. As noted in Table 21, the higher percentage of pores larger than 5 microns in diameter found in the highly porous 2.45 minus material as compared to that in the 2.45-2.55 and 2.55 plus

fractions apparently facilitated absorption in the lightweight chert with a resulting higher degree of saturation than could be obtained in the heavier materials.

As has been shown previously, chert with a specific gravity of less than 2.45 caused the only deterioration in the freeze-thaw test. From the data it appears that cherts with vacuum-saturated absorptions of less than about three per cent will not cause significant freeze-thaw deterioration when included in concrete in amounts up to ten per cent of the coarse aggregate for the size groups studied. Apparently chert with absorptions of about four per cent or greater will cause freeze-that failure when used in amounts as low as six per cent of the coarse aggregate.

The vacuum-saturated absorption values for the shales (Table 13) varied considerably, the percentages of absorption roughly paralleling the porosities of the shale samples. As was the case for porosity, the absorption of the shales had no apparent influence on the resistance of concrete containing these materials to deep-seated deterioration. This is shown by the fact that none of the shales, including the most absorptive samples, caused any deep-seated freeze-thaw failure when used in concrete in amounts up to ten per cent of the coarse aggregate.

There does appear to be a relationship between absorption and severity of surface deterioration, however. Shale 2068, which has the greatest absorption, caused by far the greatest amount of popout damage. As was the case for porosity, the influence of absorption on

surface deterioration is not so distinct among the other four shales. Shales 2066 and 2076 had considerably greater absorptions than shales 2063 and 2075 (Table 13), but there was little difference in the amount of surface deterioration caused by these four shales. Although the popout damage caused by shales 2066 and 2076 was slightly greater than that caused by shales 2063 and 2075, it was not as great as might be expected in light of the severe damage to concrete containing shale 2068. It is probable that the great difference in surface deterioration between shale 2068 and shales 2066 and 2076 is not entirely due to the greater porosity and absorption of sample 2068, but was at least partially a result of the relative lack of induration of this shale as compared to the others.

Rate of Absorption

The rates of absorption of cherts 2067 and 2077 (Figure 7) apparently had little influence on freeze-thaw durability of these materials. Material in all three specific gravity groups of chert 2067 attained nearly maximum absorption for the test in only five minutes, while the absorption of chert 2077 for the same specific gravity groups for the first five minutes was only about 25 per cent of its total absorption. Although the total porosities and absorptions of these two cherts were similar, the rates of absorption indicate the two materials have considerably different pore systems. Chert 2067 obviously is more permeable than chert 2077. This difference in permeability between cherts 2067 and 2077 is reflected in the degrees of saturation obtained by vacuum-saturating these two aggregates (Table 21). At all specific gravity levels chert 2067 had higher saturation coef-

ficients than chert 2077. In spite of this difference in permeability and its resulting difference in degree of saturation, these two cherts resulted in similar deterioration in the freeze-thaw test at all specific gravity levels.

It should be noted that the freeze-thaw tests were conducted under rather severe saturation conditions. The aggregate was vacuum-saturated before mixing the concrete, and the beams were immersed in water for 13 days prior to being subjected to freezing and thawing. They were, of course, re-immersed during each thaw cycle. Under such conditions both cherts maintained a high degree of saturation (Table 21). Under actual service conditions, however, the amount of available water would not always be as great as in these laboratory tests and permeability could have a greater influence on freeze-thaw durability.

The rates of absorption of the shales (Figure 8) are directly related to the total absorptions of these materials. Those shales with high total absorptions absorbed water rapidly during the first few minutes of the test, following which water was absorbed at a slowly decreasing rate for the rest of the test. The shales with low total absorptions exhibited a fairly constant increase in absorption throughout the test. As was the case with porosity and total absorption, this greater permeability of certain shales had no influence on the resistance of the shales to deep-seated deterioration. It probably is a factor in surface deterioration, however, since shale 2068 which caused the most popout damage also had the fastest rate of absorption.

Mineralogy

Although Runner (49), Rhoades and Mielenz,(46, 47), and Mather and Mather (31) have studied the mineralogy of deleterious aggregates, very little has been published on the relationship between mineralogy and freeze-thaw durability for these materials. Therefore part of the petrographic study of the six cherts used in this investigation is concerned with the effect of mineralogy of cherts on the freeze-thaw durability of these materials.

Microscopic petrography of the cherts has indicated their mineralogies are generally similar. One difference in mineral composition occurs in the samples from the southern part of Indiana (especially the material from the Ohio River). These contain considerably more limonite than samples from the northern part of the state. This difference in limonite content apparently had no influence on freeze-thaw durability, however, since all six cherts reacted similarly to the freeze-thaw tests.

It also is of interest to note that although there is considerable difference in freeze-thaw durability of material within the different specific gravity groups for each chert source, the mineral varieties of the different specific gravity groups are similar. There apparently is no relation between mineralogy and freeze-thaw durability of Indiana cherts.

In the past, little attempt has been made to correlate shale mineralogy and freeze-thaw durability. In this study, microscopic petrography, x-ray diffraction, and differential thermal analysis showed that all the shales are made up of essentially the same minerals, but the relative amounts of these minerals differ from one shale to

another. For example, shales 2063 and 2075, which are the most indurated and have the lowest porosities and absorptions of the samples tested, contain more detrital quartz and less clay mineral than the other shales. On the other hand, shale 2068, which is the most poorly indurated and has the highest porosity and absorption of the shale samples, contains a relatively low percentage of quartz and has a high clay mineral content. The relative percentages of quartz and clay minerals undoubtedly determine the strength characteristics of these shales, and the shale porosities apparently are directly related to clay mineral content.

Although the differences in relative percentages of clay minerals and quartz have no effect on the tendency of the shales to resist deep-seated deterioration, these mineralogic differences apparently do influence the amount of surface deterioration caused by the shales. This is especially true for shale 2068. Its high clay mineral content renders it weaker and more porous than the other shales, and thus it is more susceptible to freeze-thaw deterioration.

Texture and Microstructure

The textures and microstructures of the cherts and shales are of particular interest because it is primarily upon these properties that pore characteristics are based. The textures and microstructures of the cherts are all similar, as shown in the section on results of tests of basic properties, except that material in the 2.45 minus specific gravity range from each chert contained numerous voids large enough to be recognized in thin sections (about 30 microns or larger). These voids ranged in size up to 0.4 and 0.5 mm. in diameter, but most were

less than 0.1 mm. in diameter. As noted in the previous section on porosity, the concentration of these voids in the chert with specific gravity less than 2.45 resulted in the relatively high porosity of this lightweight chert, and the high degree of saturation achieved in the lightweight chert fractions is probably also related to the presence of these voids. Voids of this size had previously been recognized in thin sections of lightweight cherts from other states by Wuerpel and Rexford (74) who noted that these voids were related to the lack of durability of the cherts.

The 2.45-2.55 and 2.55 plus specific gravity groups contained practically no voids large enough to be recognized in thin section. Since lack of freeze-thaw durability was found only in the 2.45 minus chert, there is a direct correlation between the presence of these voids and the lack of durability of the lightweight chert. Although this contradicts the theories of Blanks (5) and others that freeze-thaw deterioration occurs primarily in voids less than 5 microns in diameter, there is a strong possibility (as demonstrated in the section on porosity) that the larger voids are prime factors in the freeze-thaw breakdown of lightweight cherts due to the higher degree of saturation afforded the chert by the larger voids.

Other textural properties such as grain size, and presence of rhombic-shaped grains and replaced fossils, apparently had no influence on the freeze-thaw durability of the chert. These characteristics are similar in cherts of all three specific gravity ranges.

Again in the shales, the texture and microstructure influenced the freeze-thaw durability of these materials only in that pore

structure is closely controlled by these properties. In general, for the shales studied, it was found that the finer-grained shales, i.e., those containing large amounts of clay minerals, had greater porosities than the coarser ones.

Apparently the technique used by Mitchell (36) for determining relative degrees of orientation of clay particles is too qualitative for indicating relative porosity or predicting freeze-thaw durability of shales. In spite of the widely varying porosities and different degrees of surface deterioration caused by these shales, all five shales appeared to have about the same amount of parallelism of grains.

ACCORDANCE OF RESULTS WITH SPECIFICATIONS ON DELETERIOUS MATERIALS IN CONCRETE AGGREGATES

One of the primary purposes of this study was to determine whether the present (1957) specifications of the State Highway Department of Indiana (55) on deleterious substances in aggregates categorize these materials on a realistic basis. This study was organized so that the effects of several variables on freeze-thaw durability of cherts and shales in conrete could be noted and related to the present specifications.

The Indiana specifications allow a maximum of three per cent of chert with a bulk specific gravity (saturated surface-dry basis) of less than 2.45. The results of the freeze-thaw tests have shown that the establishment of 2.45 as a critical specific gravity level separating sound and unsound cherts is completely justified.

The specifications do not single out shale, but include it with "soft and non-durable particles," i.e., particles which are structurally weak such as soft sandstone, shale, limonite concretions, coal, weathered schist, and cemented gravel. The total weight of these materials should not exceed four per cent of the coarse aggregate. This means that shale is allowed in amounts up to four per cent depending on the quantity of the other soft and non-durable materials present. The controlling factor in establishing four per cent as the critical level has been structural strength of the concrete, not freeze-thaw durability. The results of this study indicate that as far as deep-seated freeze-thaw deterioration of concrete is concerned, the four per cent restriction

is probably safe. However, the freeze-thaw studies have shown that significant surface deterioration occurs in concrete containing certain soft, porous shales in amounts as low as two per cent. Therefore, even when this specification (a total of four per cent of soft particles) is rigidly adhered to, some popout damage should be expected from the most porous shales.

SUMMARY OF RESULTS

The following is a brief recapitulation of major findings of the study:

1. Freezing-and-thawing tests of concrete beams containing chert indicated the following:

   (a) There was no difference in degree of deep-seated deterioration caused by any of the different cherts. Thus the source of chert had no effect on freeze-thaw durability.

   (b) The only combination of variables resulting in severe deep-seated deterioration was ten per cent of 2.45 minus chert. This combination resulted in deep-seated failure of all beams containing chert from each of the six sources. In addition, six per cent of 2.45 minus chert caused moderate deep-seated damage in a few cases.

2. Durability factors for the shale beams indicated that no deep-seated deterioration occurred in beams containing two to ten per cent of any of the five shales studied. The data included no extremely low durability factors as were found for beams containing ten per cent of 2.45 minus specific gravity chert. Only a few beams had durability factors below 90, and these few values were seemingly randomly distributed throughout the data.

3. Study of surface deterioration of concrete beams containing chert showed that freezing and thawing caused significant popout damage in beams containing 2.45 minus specific gravity chert. Very few popouts were caused by chert having specific gravities of 2.45-2.55 and 2.55 plus.

4. The greatest amount of surface deterioration of the beams containing shale was caused by shale 2068, the most porous and most absorbent of the shales. Shale 2068 caused considerable popout and pitting damage at all four percentage levels, but, as would be expected, the amount of deterioration increased with increasing percentage of shale. The other four shales tested caused no surface deterioration when included in concrete in amounts up to and including four per cent. At the six and ten per cent levels, shales 2066 and 2076, which were more porous and absorbent than shales 2063 and 2075, caused slightly more surface deterioration than the latter.

5. The study of air voids in concrete by means of the linear traverse technique demonstrated that machine-mixed concrete beams with high durability factors had air-void spacing factors lower than 0.01 inches, and hand-mixed beams with low durability factors had spacing factors higher than 0.01 inches. These results support Powers' theory that concretes with spacing factors lower than 0.01 inches are well protected from freezing and thawing deterioration, while those with spacing factors greater than 0.01 inches are poorly protected.

6. The porosities of the cherts studied ranged from about 3 per cent for the chert fractions with bulk specific gravities (saturated

surface-dry basis) of 2.55 plus to almost 13 per cent for the 2.45 minus material.

The porosities of the shales varied widely. The two well-indurated, non-fissile shales, samples 2063 and 2075, had the lowest porosities of the samples tested, 4.2 per cent and 8.6 per cent, respectively. Shales 2066 and 2076, which were intermediate in strength and hardness, had intermediate porosities, 13.8 per cent and 15.7 per cent respectively. The softest and weakest shale, 2068, had the highest porosity, 22.5 per cent.

7. Study of the size distributions of pores for cherts 2067 and 2077 indicated a marked increase in percentage of total voids volume consisting of pores larger than 5 microns in diameter with decreasing bulk specific gravity of the chert. For example, in the case of chert 2067, the voids larger than 5 microns in diameter constituted only 20 per cent of the total pore space in the 2.55 plus chert, 25 per cent in the 2.45-255 material, and 50 per cent in the 2.45 minus range. Conversely, the voids less than 5 microns in diameter constituted a decreasing percentage of total voids volume with increase in total porosity and resulting decrease in bulk specific gravity.

8. The degree of saturation achieved by vacuum immersion of cherts 2067 and 2077 increased with increasing total porosity and decreasing bulk specific gravity. For example, the coefficient of saturation for chert 2067 was 82 per cent for the 2.55 plus material, 93 per cent for the 2.45-2.55 chert, and approximately 100 per cent

for the material in the 2.45 minus specific gravity range.

9. In general, the absorption characteristics of cherts from all sources tested were similar. As expected, the absorptions of the low bulk specific gravity fractions were higher than those of the high specific gravity groups. For all six cherts, material in the 2.45 minus fractions had absorption percentages about twice as great as those for the 2.45-2.55 groups and five times as great as the 2.55 plus groups. The absorption values ranged from about one per cent for the 2.55 plus material to about five per cent for the 2.45 minus groups.

The absorption values for the shales varied widely among the five sources, roughly paralleling the porosities of the shale samples. Shale 2063, the least porous sample, had an average vacuum-saturated absorption of only 1.8 per cent, while shale 2068, the most porous sample, had an average absorption of 12.6 per cent. The other three shale samples had absorption values between these two extremes.

10. Although the total porosities and absorptions of cherts 2067 and 2077 were similar, their rates of absorption indicate that these two cherts have considerably different pore systems. Chert 2067 was more permeable than chert 2077. Material in all three specific gravity groups of chert 2067 attained nearly maximum absorption after only five minutes of immersion, while the absorption of the same specific gravity groups of chert 2077 for the first five minutes was only about 25 per cent of its total absorption.

The rates of absorption of the shales were directly related to

the total absorptions. Those shales with high total absorptions absorbed water rapidly during the first few minutes of immersion, following which water was absorbed at a slowly decreasing rate for the rest of the test. The shales with low total absorptions exhibited a fairly constant increase in absorption throughout the test.

11. Petrographic analysis of thin sections showed the cherts to be of generally similar mineralogical character. They were composed primarily of microcrystalline quartz and radial chalcedony. Small amounts of coarse-grained secondary quartz, some calcite, and limonite were also present. One chief mineralogical difference was noted. Cherts from the southern part of Indiana (especially from the Ohio River) contained more limonite than those from the northern part of the state. This limonite occurred both as rhombs and in amorphous form. No differences in mineralogy were noted among the three specific gravity groups for the chert samples.

The shales also presented a similarity in their general mineralogic compositions, but showed considerable variability in certain characteristics. All the shales consisted of detrital mineral grains, primarily quartz, in a very fine-grained matrix of clay minerals or hydromicas. The chief differences shown by the shales were the relative size and abundance of the detrital mineral grains and the relative amounts of clay minerals and organic material in the samples.

12. The textures and microstructures of the cherts were all similar. Each chert consisted primarily of microcrystalline aggregates of quartz grains usually less than 0.01 mm. in diameter with granular masses of secondary quartz, radiating masses of chalcedony,

and carbonate and limonite rhombs. The only notable structural difference in the cherts was that the 2.45 minus fraction of each sample contained numerous voids large enough to be identified in thin sections between crossed nicols. These voids, which averaged less than 0.1 mm. in size, but ranged in size up to 0.4-0.5 mm., did not occur in the 2.55 plus and 2.45-2.55 specific gravity groups.

The textures and microstructures of the shales varied considerably. Although all the shales consisted of a fine-grained matrix enclosing detrital quartz grains, the relative amounts of these materials and the sizes of the detrital particles varied enough to influence strongly the strength and hardness of the different shales. All the shales showed preferred orientation of grains.

13. Heavy-liquid separation of the chert samples showed that all six cherts had similar specific gravity distributions. About 50 per cent of each sample fell within the 2.45-2.55 bulk specific gravity range (saturated surface-dry basis). The 2.55 plus material constituted from 15-30 per cent of each sample, and about 20-30 per cent of each sample had a specific gravity lower than 2.45.

Although the bulk of material in each of the shale samples fell in the 2.05-2.45 specific gravity range, the shale samples showed greater variability in their specific gravity distributions than did the cherts. The shales varied in specific gravity from samples 2075 and 2063 with over 70 per cent heavier than 2.25 to shales 2068 and 2066 with less than ten per cent heavier than 2.25.

CONCLUSIONS

Based on the results of this study, the following conclusions seem justified. Since this study was restricted to certain Indiana cherts and shales subjected to specific methods of test, the conclusions can logically be applied only to similar cherts and shales under similar conditions. However, in some cases, field behavior of the cherts and shales may be inferred from these conclusions.

1. For a wide variety of cherts, the source of the chert has no effect on its freeze-thaw durability in concrete.

2. Chert exhibits a definite relationship between its bulk specific gravity and durability in concrete exposed to freezing and thawing. Apparently only chert with a bulk specific gravity of less than 2.45 (saturated surface-dry basis) will cause either deep-seated or surface deterioration of air-entrained concrete in which it is used.

3. The freeze-thaw durability of concrete containing chert apparently is not as dependent on pores in the chert less than 5 microns in diameter as has been postulated by Sweet (59). Instead chert durability is apparently based on a more complicated interrelationship between total porosity, size of pores, absorption, and degree of saturation. Pores larger than the 5-micron size specified by Sweet permit easier passage of water into immersed aggregates, result in

relatively high degrees of saturation, and contribute to freeze-thaw deterioration of lightweight chert. Microscopic studies of polished sections show that these larger pores make up about half the void volume in 2.45 minus specific gravity chert.

4. The petrographic characteristics of the cherts influence the freeze-thaw durability of these materials only in the relationship of these characteristics to porosity of the cherts. For example, although mineralogy of the cherts has no direct effect on their freeze-thaw durability, the presence of carbonate rhombs, which have weathered out to form voids, has lessened the durability of some chert particles.

5. Many shales will not cause deep-seated deterioration of air-entrained concrete beams subjected to laboratory freezing and thawing when included in these beams in amounts up to ten per cent. The inability of these shales to cause deep-seated deterioration is probably due to the inherent structural weakness of these materials.

6. Different shales cause considerably different degrees of surface deterioration of air-entrained concrete exposed to freezing and thawing. Some shales cause considerable popout damage when included in concrete in amounts as low as two per cent of the coarse aggregate. Other shales cause little damage when used in amounts up to ten per cent.

7. The durability of the shales studied apparently is related primarily to the porosities and absorptions of these materials; the most porous and most absorbent causing the greatest amount of surface

deterioration of concrete in which these materials are used. However, the strength and induration of the shales, as determined by relative amounts of clay minerals and detrital quartz present, also influence the ability of these materials to cause surface deterioration, the softer, weaker materials being less resistant than the harder, stronger ones.

8. As theorized by Powers (40), concretes with air-void spacing factors lower than 0.01 inches are well-protected from freezing-and-thawing deterioration, while those with spacing factors greater than 0.01 inches are poorly protected.

SUGGESTIONS FOR FURTHER RESEARCH

Suggested further research can be summarized as follows:

1. This study could be extended to include cherts and shales from throughout the United States. In this way it would be possible to determine the effects on concrete durability of cherts and shales of more widely varying character than the Indiana cherts and shales used in this study. For a broad study of this type, it would be best to investigate the basic physical properties of several cherts and shales obtained from widely scattered gravel deposits, and then select a few samples with considerably different properties for freeze-thaw studies in concrete.

2. Since the broad studies undertaken in this investigation have indicated the pore characteristics of cherts and shales to be of prime importance in determining their freeze-thaw durabilities, a more thorough investigation of these pore characteristics utilizing the techniques used by Lewis and Dolch (27) and Dolch (11) for study of pore characteristics of limestones is indicated. A controlled study of the effect of pore size could yield valuable information.

3. Since different Indiana shales resulted in different degrees of surface deterioration of concrete, a more fully controlled investigation of "popout" damage caused by shales would be of interest. In

such a study the positioning of the individual pieces of shale could be added as a third variable, the effects of which could be analyzed along with the effects of different sources and different percentages of shale. Instead of allowing random positioning of the individual pieces of shale they would be placed at predetermined spacings and depths. As a part of this study, it would be of interest to divide some of the shale samples into bulk specific gravity fractions, and use the individual fractions in concrete subjected to freezing and thawing in order to better determine the relationship between shale porosity and surface deterioration.

4. A controlled study of surface deterioration due to freeze-thaw breakdown of cherts alao would be of interest. As in the case of the shales, the spacing and depth of the individual chert pieces would be predetermined. If confined to Indiana cherts, this test probably could be restricted to material having a bulk specific gravity (saturated surface-dry basis) of less than 2.45. If the scope of the study were broadened to include other cherts, the test should be conducted on the three gravity groups used in this investigation.

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APPENDIX A

Sample No.	C	Igneous	Meta-morphic
2063	Ada	9	4
2064	Elk	23	15
2065	War	17	13
2066	Jac	10	9
2067	Tip	27	10
2068	La F	2	2
2069	Dea	9	3
2070	Faye	9	6
2071	Madi	12	6
2072	Owen	18	3
2074	Miam	25	7

Table 22

Percentage by Weight of Rock Types in Eleven Gravel Sources

Sample No.	County	Location	Type of Glacial Deposit	Lime-stone	Dolo-mite	Sand-stone	Silt-stone	Chert	Shale	Limonite Concretions	Igneous	Meta-morphic
2063	Adams	Sec. 28, T28N, R14E	Valley Train	52	28	<1	<1	3	2	--	9	4
2064	Elkhart	Sec. 1, T37N R4E	Valley Train	29	15	4	2	12	<1	<1	23	15
2065	Warren	Sec. 27, T20N, R9W	Valley Train	52	4	2	—	11	1	--	17	13
2066	Jackson	Sec. 13, T6N, R5E	Valley Train	46	2	4	<1	26	2	<1	10	9
2067	Tippecanoe	Sec. 30, T23N, R4W	Valley Train	46	4	3	—	9	<1	<1	27	10
2068	La Porte	Sec. 33, T37N, R3W	Kame Moraine	<1	<1	8	16	<1	71	--	2	2
2069	Dearborn	Sec. 11, T5N, R1W	Valley Train	80	3	<1	—	4	<1	<1	9	3
2070	Fayette	Sec. 36, T14N, R12E	Valley Train	75	3	3	—	3	<1	—	9	6
2071	Madison	Sec. 4, T19N, R8E	Valley Train	72	4	1	—	4	<1	<1	12	6
2072	Owen	Sec. 6, T10N, R2W	Valley Train	65	2	2	<1	8	<1	<1	18	3
2074	Miami	Sec. 25, T27N, R5E	Valley Train	54	4	2	—	7	<1	—	25	7

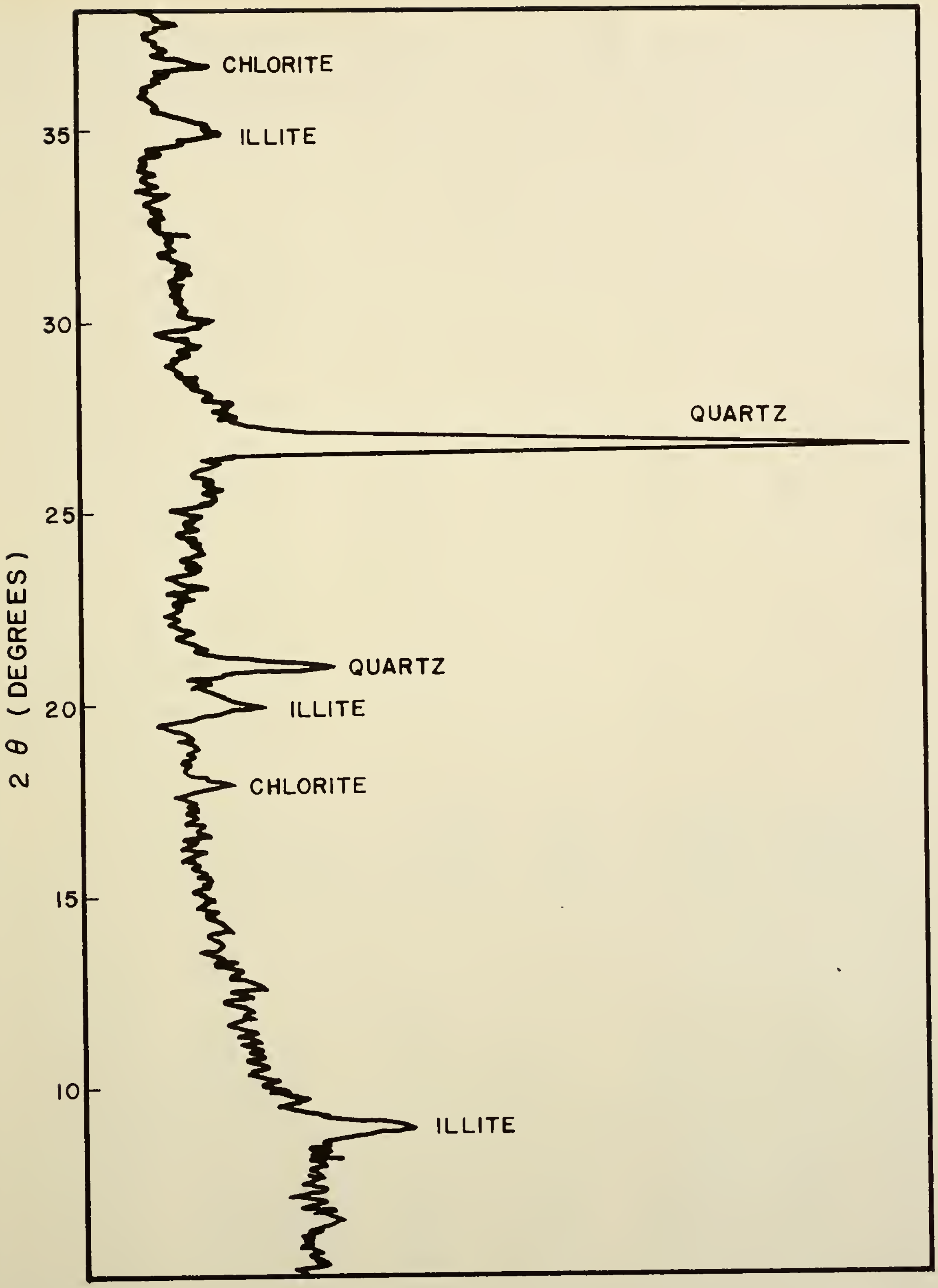

FIGURE 20. X-RAY SPECTROMETER TRACE FOR SHALE 2076

Table 23

Summary of Results of Petrographic Studies of Shales

Shale	Degree of Induration	Abundant Mineral Constituents	Grain Size of Clay Mineral Matrix (mm.)	Avg. Size of Detrital Quartz Grains (mm.)	Max. Size of Detrital Grains (mm.)	Loss on Ignition (per cent)
2063 (Decatur)	high	illite quartz limonite chlorite	< 0.01	0.02-0.03	0.07	16.7
2066 (Seymour)	medium	hydromica quartz limonite chlorite	< 0.01	0.01-0.02	0.04	16.5
2068 (near LaPorte)	low	illite limonite quartz chlorite	< 0.01	0.01-0.02	0.04	9.2
2075 (Indianapolis)	high	illite quartz limonite chlorite	< 0.01	0.01-0.02	0.04	12.3
2076 (South Bend)	medium	illite quartz limonite chlorite	< 0.01	0.01-0.02	0.04	11.6

APPENDIX B

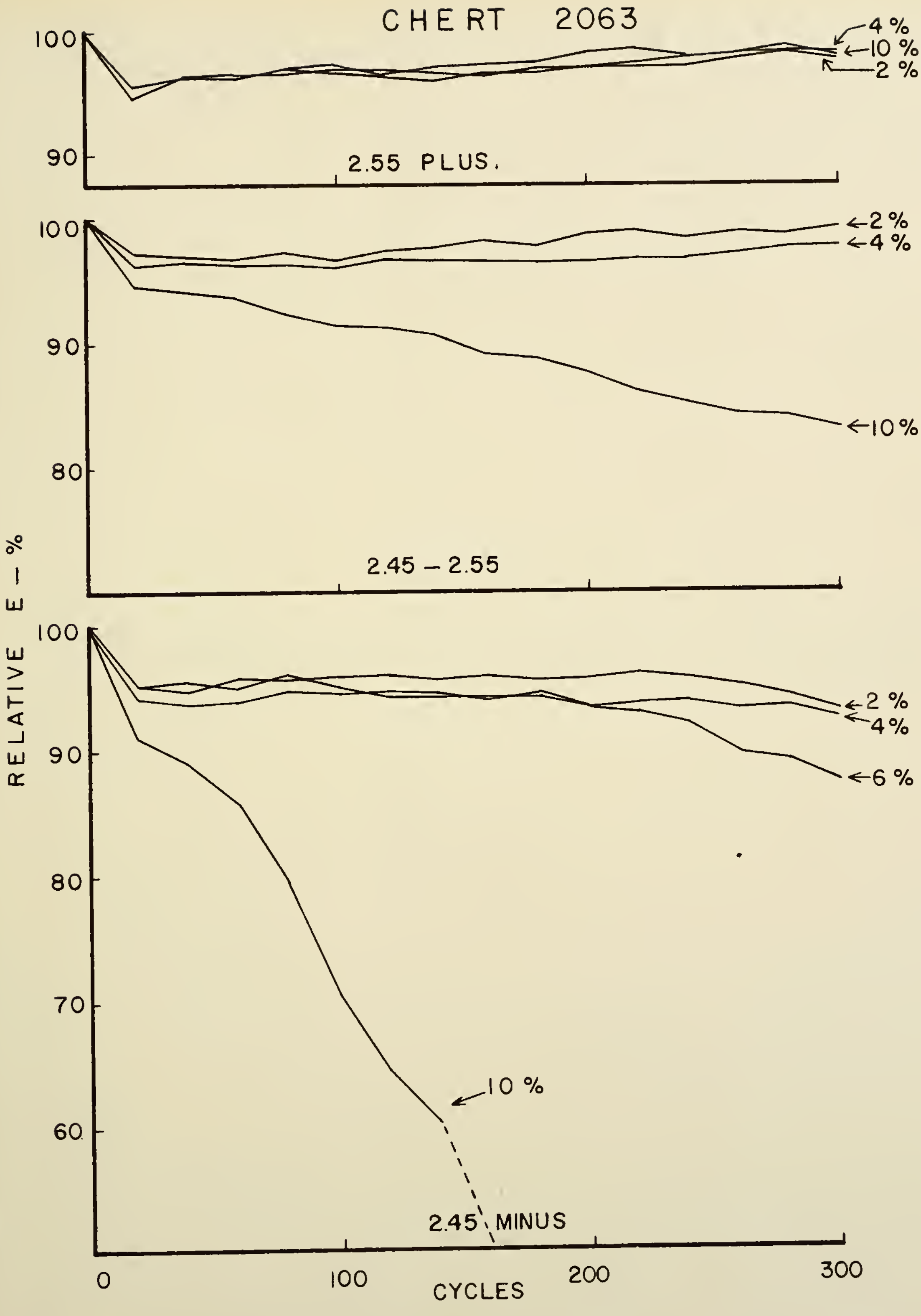

FIGURE 21. AVERAGE FREEZE-THAW CURVES FOR CONCRETE BEAMS CONTAINING CHERT 2063

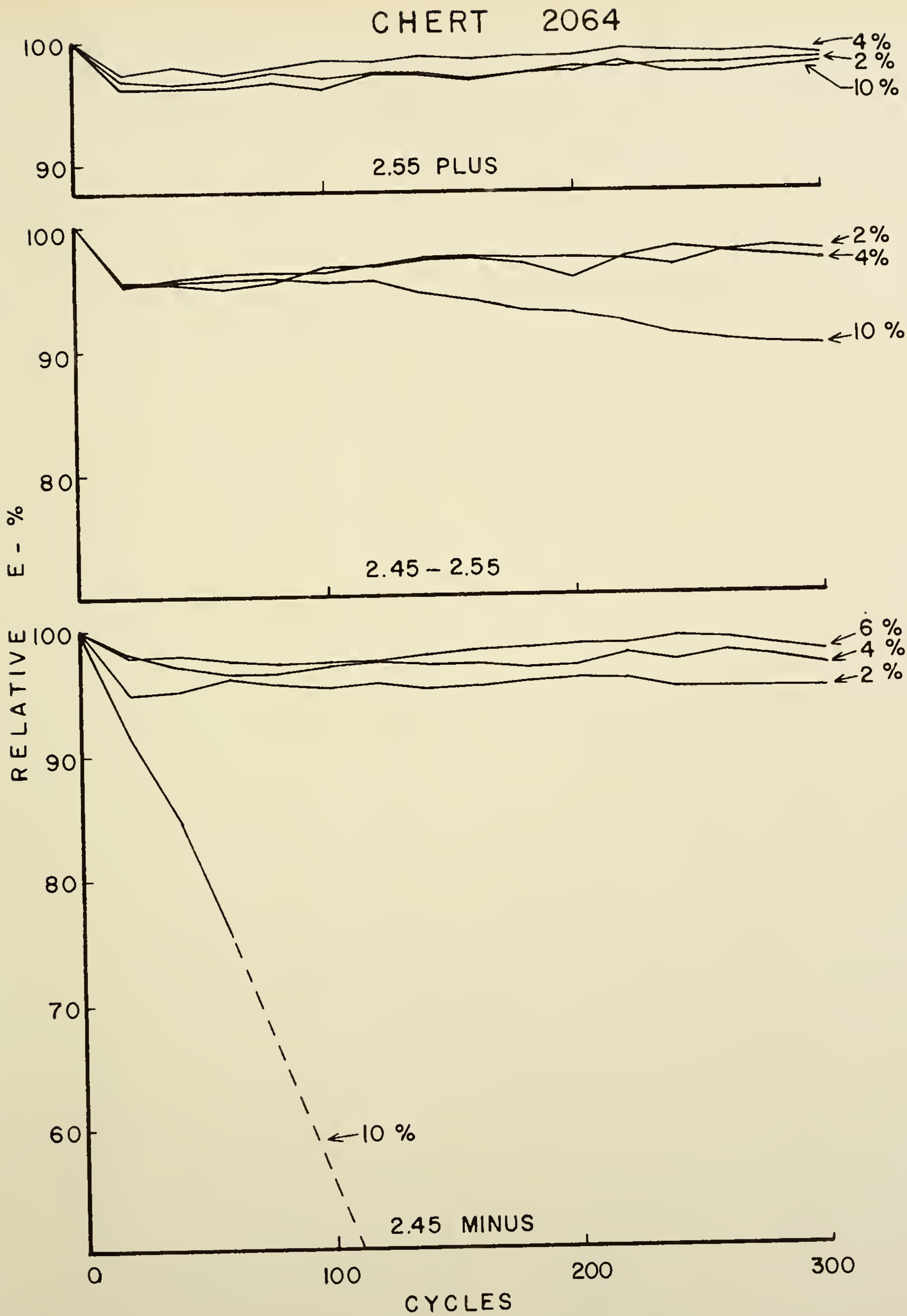

FIGURE 22. AVERAGE FREEZE-THAW CURVES FOR CONCRETE BEAMS CONTAINING CHERT 2064

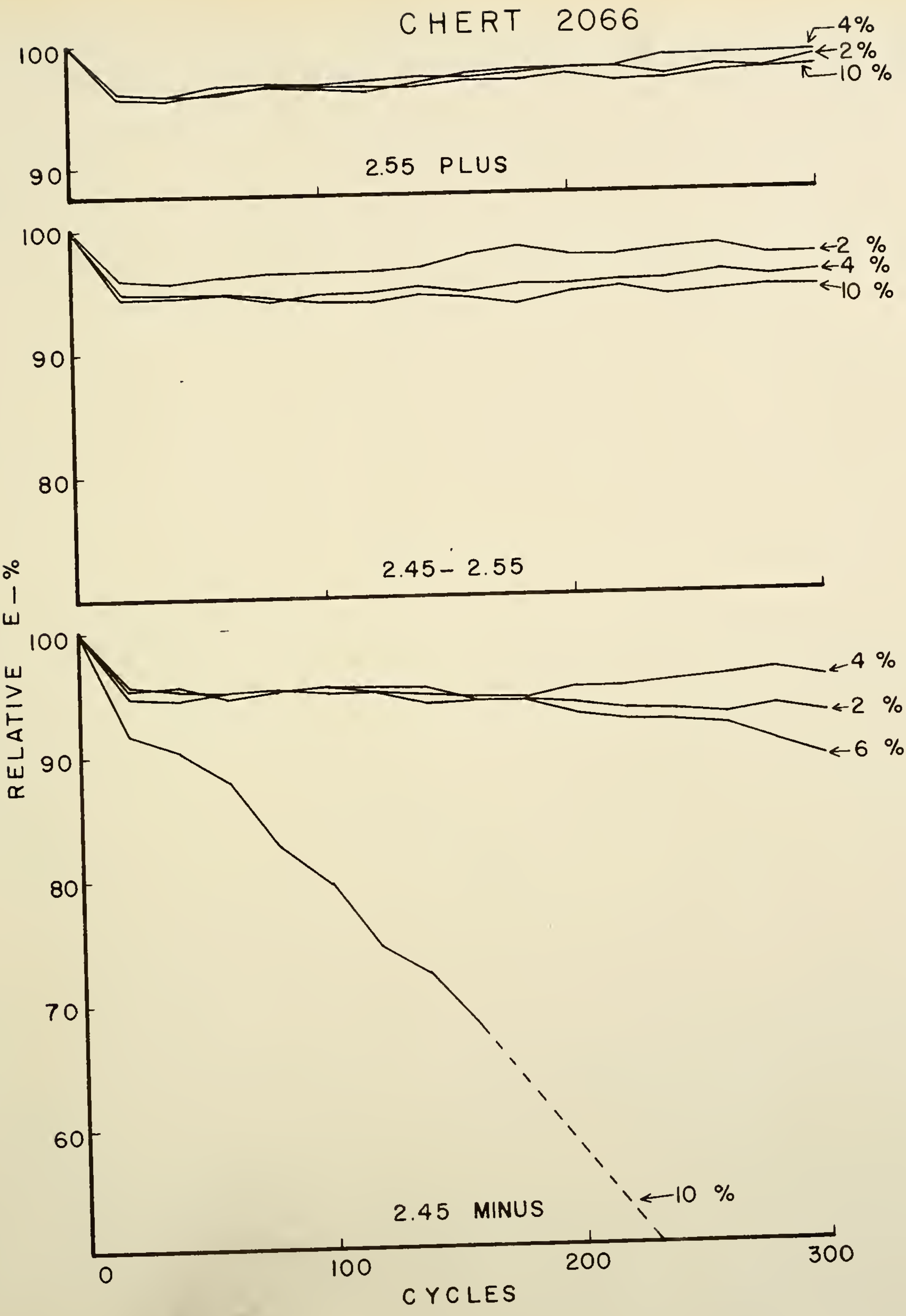

FIGURE 23. AVERAGE FREEZE-THAW CURVES FOR CONCRETE BEAMS CONTAINING CHERT 2066

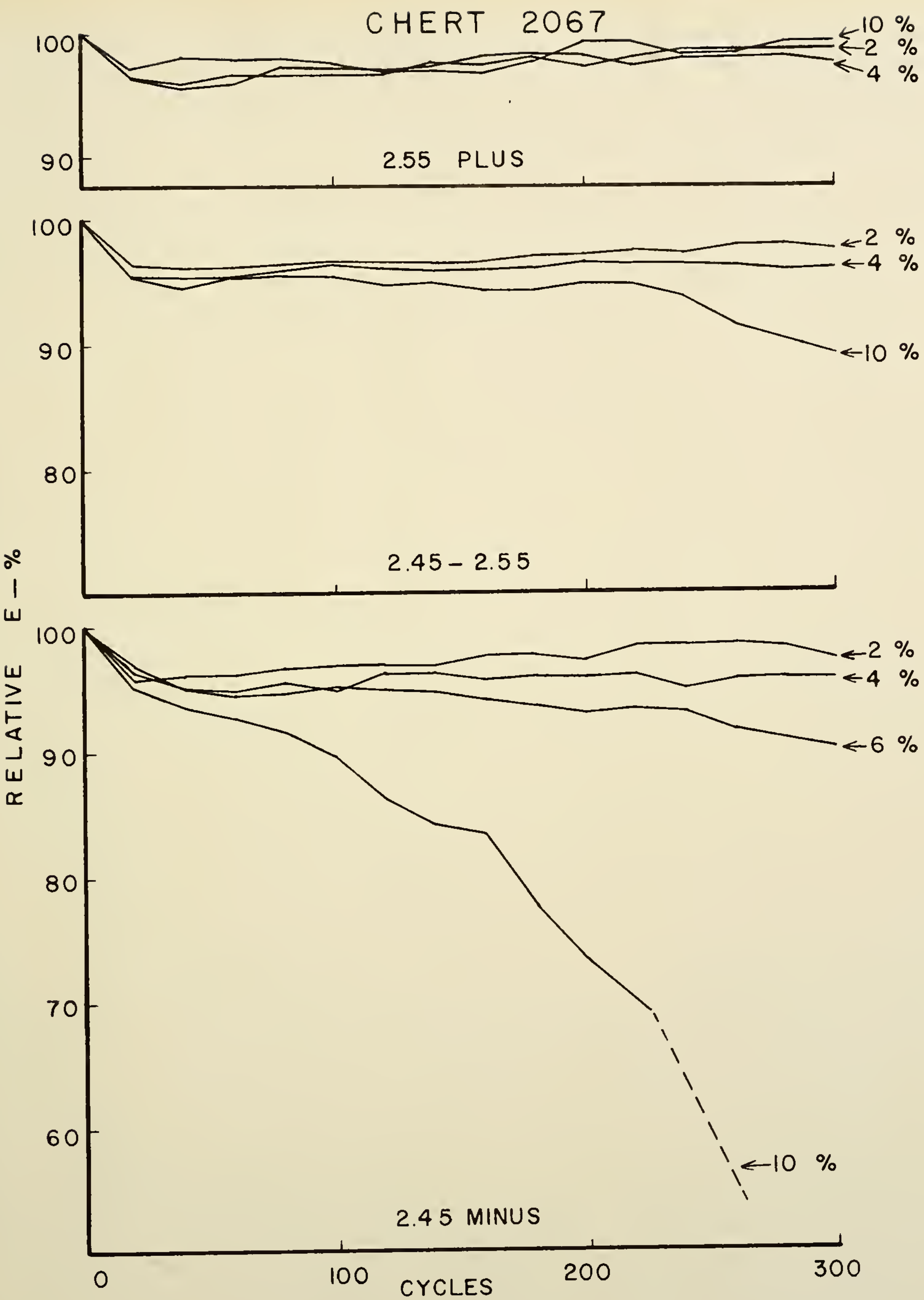

FIGURE 24. AVERAGE FREEZE-THAW CURVES FOR CONCRETE BEAMS CONTAINING CHERT 2067

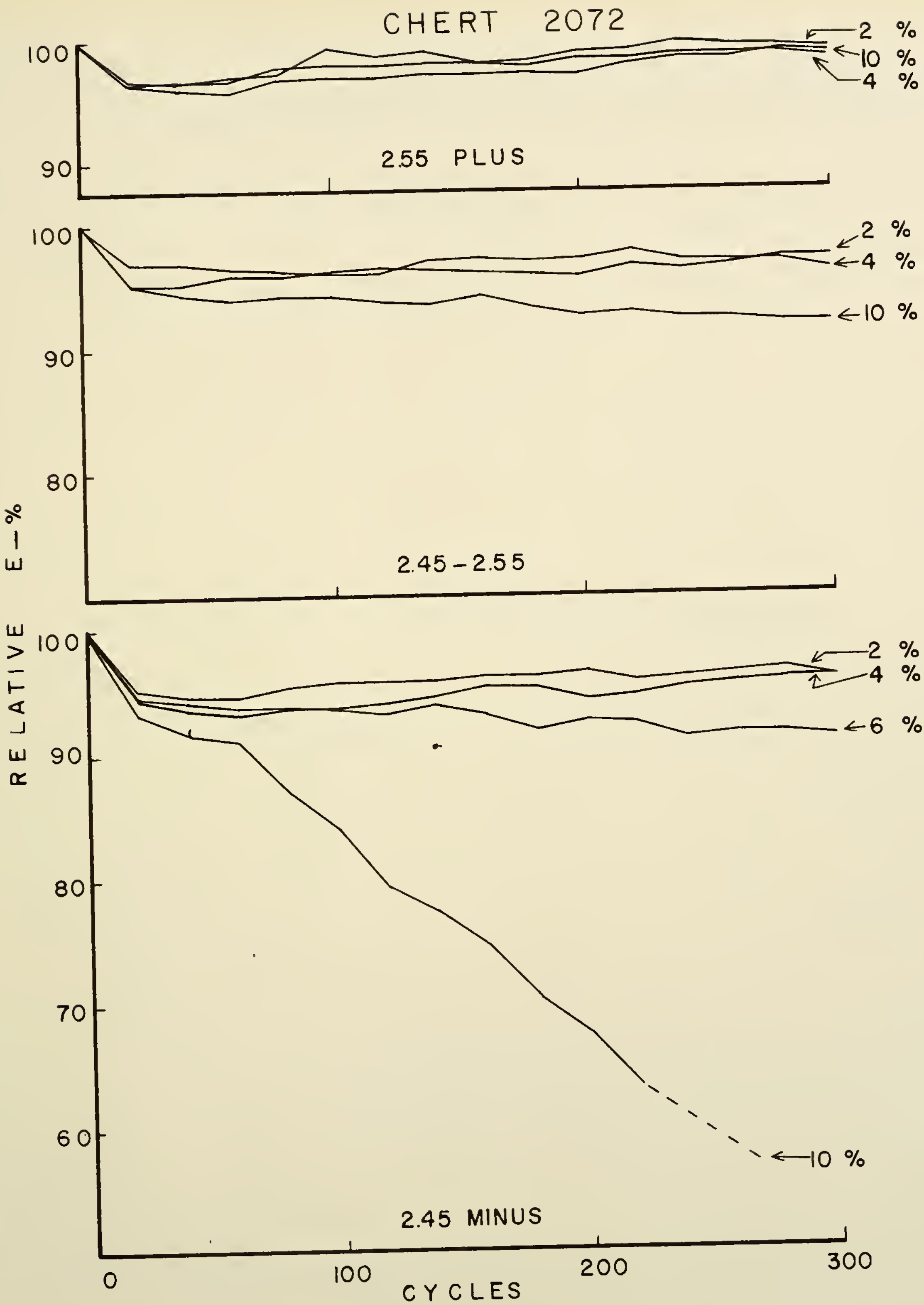

FIGURE 25. AVERAGE FREEZE-THAW CURVES FOR CONCRETE BEAMS CONTAINING CHERT 2072

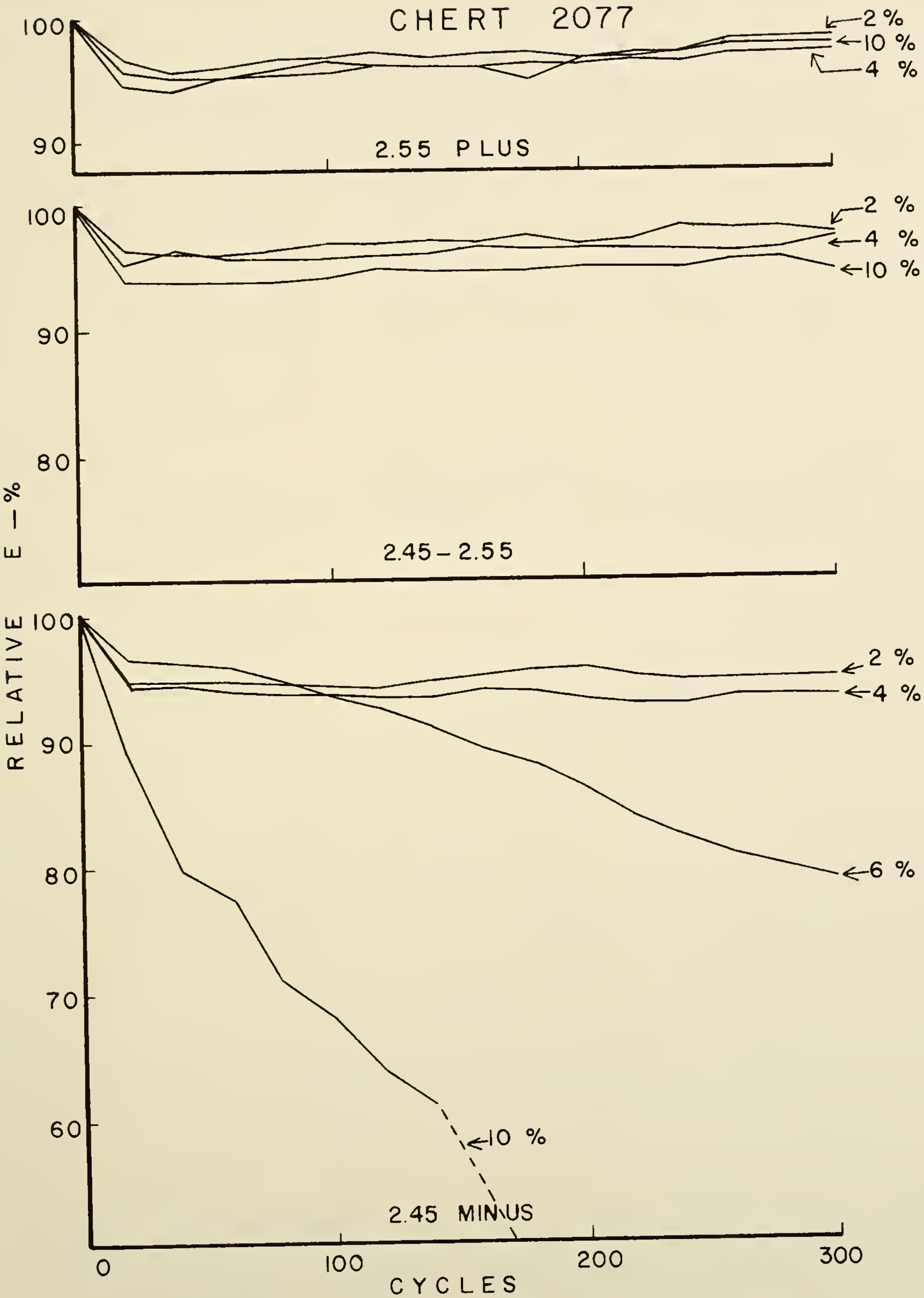

FIGURE 26. AVERAGE FREEZE-THAW CURVES FOR CONCRETE BEAMS CONTAINING CHERT 2077

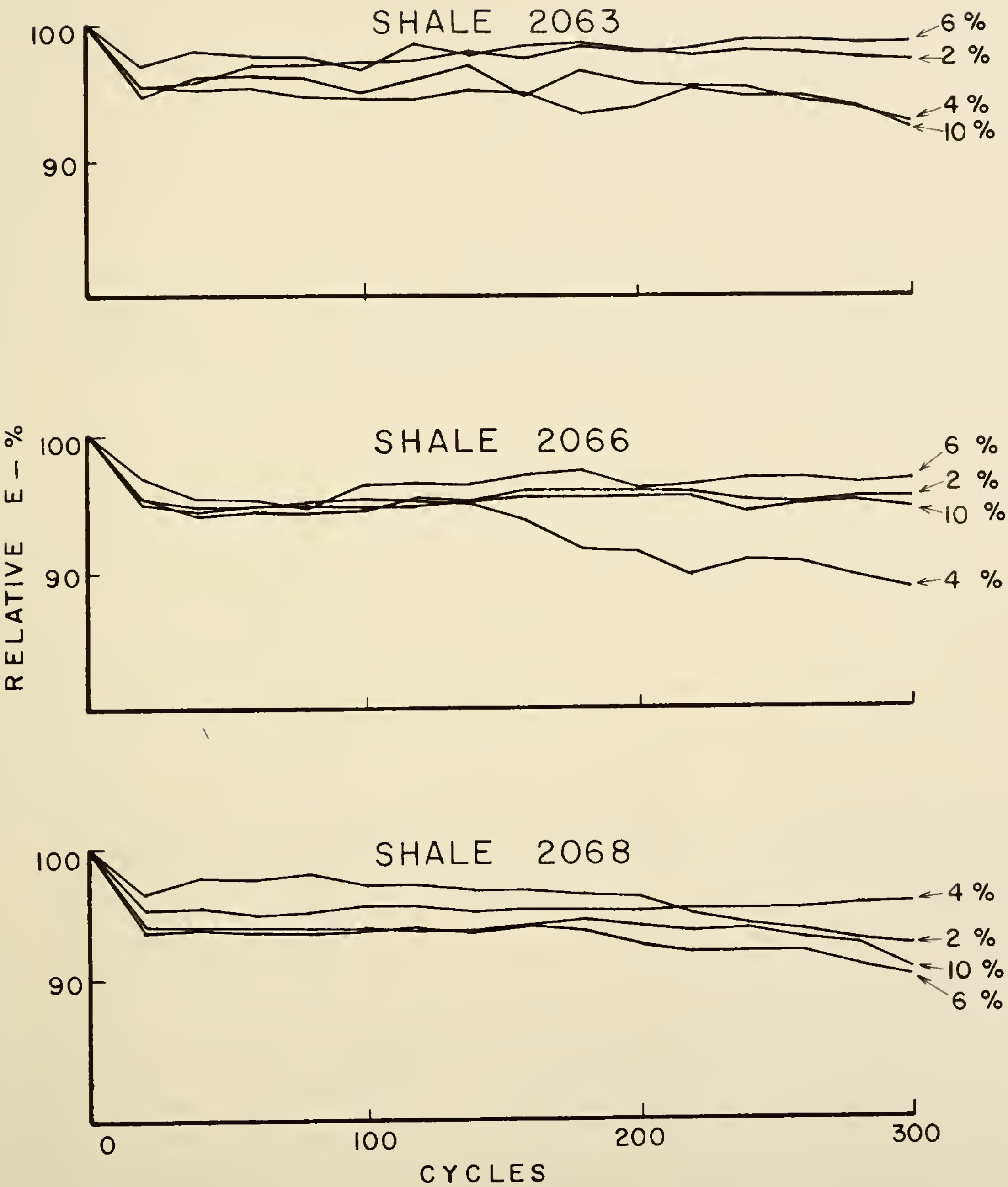

FIGURE 27. AVERAGE FREEZE-THAW CURVES FOR CONCRETE BEAMS CONTAINING SHALES 2063, 2066, AND 2068

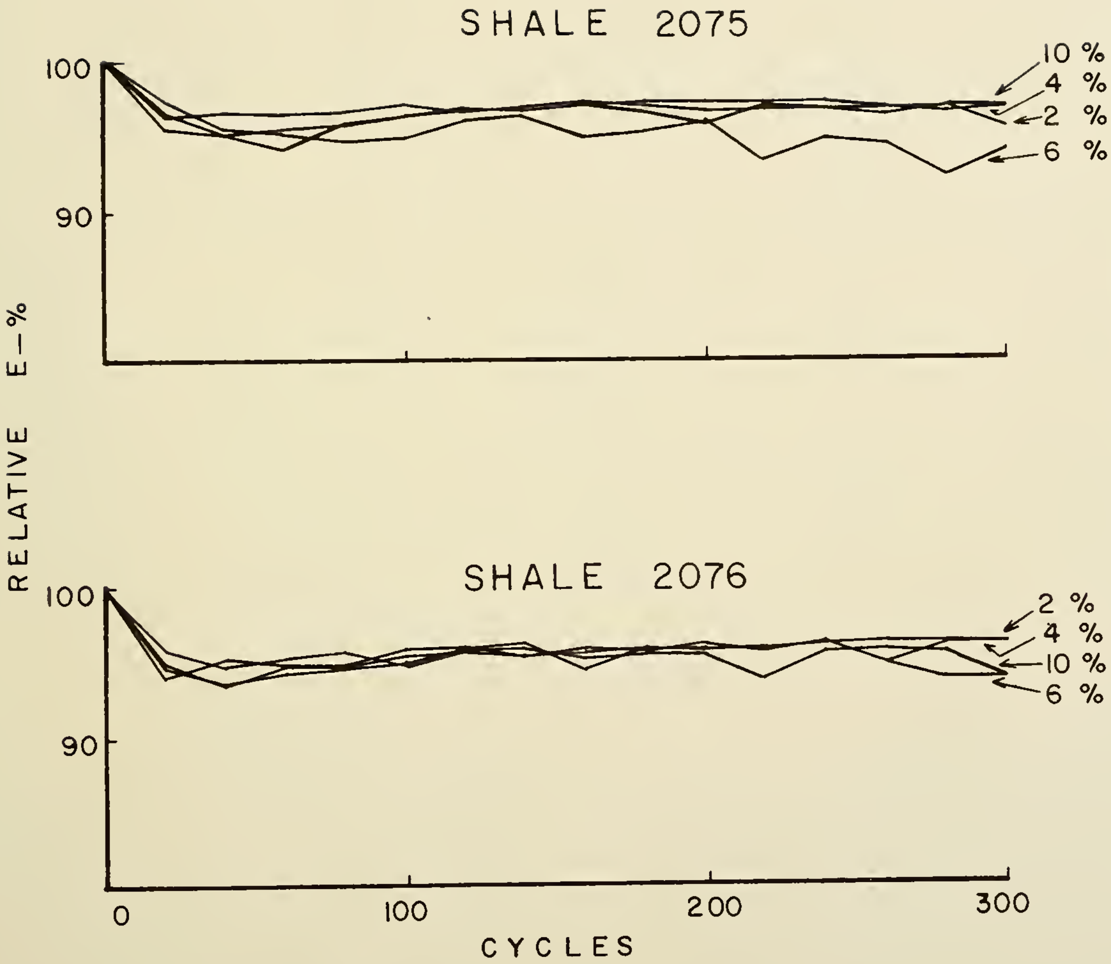

FIGURE 28. AVERAGE FREEZE-THAW CURVES FOR CONCRETE BEAMS CONTAINING SHALES 2075 AND 2076

Table 24

Durability Factors Deleted From Table 16

Per Cent Shale in Coarse Aggregate	Shale Source: 2063 (Decatur)	2066 (Seymour)	2068 (near LaPorte)	2075 (Indianapolis)	2076 (South Bend)
2%	--	62.3	--	48.9	--
4%	--	78.5 26.3	43.0 44.2	--	--
6%	--	--	42.4 44.6 41.7	--	--
10%	--	33.8	27.2 47.6	52.0	--

Table 25

Surface Deterioration Factors Deleted from Table 18

Per Cent Shale in Coarse Aggregate	Shale Source: 2063 (Decatur)	2066 (Seymour)	2068 (near LaPorte)	2075 (Indianapolis)	2076 (South Bend)
2%	--	0.0	--	0.0	--
4%	--	2.0	2.5	--	--
6%	--	--	1.3 3.3 5.0	--	--
10%	--	0.5	2.7 6.0	1.0	--

Table 26

Comparison of Mean Durability Factors for Beams Containing Ten Per Cent to Those Containing Two and Four Per Cent of 2.55 Plus Specific Gravity Chert

---

Hypotheses:

$$H_o : \mu_1 = \mu_2$$

$$H_1 : \mu_1 > \mu_2$$

<u>Level of Significance</u>:

$$\alpha = 0.01$$

<u>Fisher-Behrens Test</u>:

<u>Two and Four Per Cent Levels</u>	<u>Ten Per Cent Level</u>
$n_1 = 24$	$n_2 = 12$
$s^{2*}_{\bar{x}_1} = 0.026$	$s^2_{\bar{x}_2} = 0.083$
$\bar{X}_1 = 98.08$	$\bar{X}_2 = 98.02$

---

* For all comparison testing in this paper, $s^2_{\bar{x}}$ was calculated as follows:

$$s^2_{\bar{x}} = \frac{\sum_{i=1}^{n/2} R_i^2}{n^2}$$

Table 26 (continued)

Comparison of Mean Durability Factors for Beams Containing Ten Per Cent to Those Containing Two and Four Per Cent of 2.55 Plus Specific Gravity Chert

$$t_{obs.} = \frac{98.08 - 98.02}{\sqrt{0.026 + 0.083}} = 0.18 \qquad t_{18.3,\ 0.02}^{*} = 2.58$$

$0.18 < 2.58$ Therefore accept H_o .

* Degrees of freedom for the t-distribution were calculated as follows:

$$\frac{1}{\gamma} = \frac{1}{\gamma_1}\left(\frac{s^2_{\bar{x}_1}}{s^2_{\bar{x}_1} + s^2_{\bar{x}_2}}\right)^2 + \frac{1}{\gamma_2}\left(\frac{s^2_{\bar{x}_2}}{s^2_{\bar{x}_1} + s^2_{\bar{x}_2}}\right)^2$$

where

γ = degrees of freedom for the t-distribution

$\gamma_1 = n_1 - 1$

$\gamma_2 = n_2 - 1$

Table 27

Comparison of Mean Durability Factors for Beams Containing Ten Per Cent to Those Containing Two and Four Per Cent of 2.45-2.55 Specific Gravity Chert

---

Hypotheses:

$H_o : \mu_1 = \mu_2$

$H_1 : \mu_1 > \mu_2$

Level of Significance:

$\alpha = 0.01$

Fisher-Behrens Test:

Two and Four Per Cent Levels	Ten Per Cent Level
$n_1 = 24$	$n_2 = 12$
$s^2_{\bar{x}_1} = 0.036$	$s^2_{\bar{x}_2} = 6.26$
$\bar{X}_1 = 97.10$	$\bar{X}_2 = 90.51$

$$t_{obs.} = \frac{97.10 - 90.51}{\sqrt{0.036 + 6.26}} \qquad t_{10.8,\ 0.02} = 2.77$$

$2.63 < 2.77$ Therefore accept H_o

Table 28

Comparison of Mean Durability Factors for Beams Containing Ten Per Cent to Those Containing Two, Four, and Six Per Cent of 2.45 Minus Specific Gravity Chert

---

Hypotheses:

$H_o : \mu_1 = \mu_2$

$H_1 : \mu_1 > \mu_2$

Level of Significance:

$\alpha = 0.01$

Fisher-Behrens Test:

Two, Four, and Six Per Cent Levels

$n_1 = 36$

$s^2_{\bar{x}_1} = 0.78$

$\bar{X}_1 = 92.77$

Ten Per Cent Level

$n_2 = 12$

$s^2_{\bar{x}_2} = 10.90$

$\bar{X}_2 = 35.11$

$$t_{obs.} = \frac{92.77 - 35.11}{\sqrt{10.90 + 0.78}} = 16.8 \qquad t_{12.6,0.02} = 2.70$$

$16.8 > 2.70$ Therefore reject H_o

Table 29

Comparison of Mean Durability Factors for Beams Containing Six Per Cent to Those Containing Two and Four Per Cent of 2.45 Minus Specific Gravity Chert

---

Hypotheses:

H_o : $\mu_1 = \mu_2$

H_1 : $\mu_1 > \mu_2$

Level of Significance:

$\alpha = 0.01$

Fisher-Behrens Test:

Two and Four Per Cent Levels	Six Per Cent Level
$n_1 = 24$	$n_2 = 12$
$s^2_{\bar{x}_1} = 0.33$	$s^2_{\bar{x}_2} = 5.72$
$\bar{X}_1 = 94.84$	$\bar{X}_2 = 88.63$

$$t_{obs.} = \frac{94.84 - 88.63}{\sqrt{5.72 + 0.33}} = 2.54 \qquad t_{12.1,\ 0.0.02} = 2.72$$

$2.54 < 2.72$ Therefore accept H_o.

APPENDIX C

Derivation of Equation for Total Porosity

By definition:

$$n = \frac{V_v}{V}$$

where

n = porosity,

V_v = volume of voids,

V = total volume of solids and voids.

Therefore

$$n = \frac{V_v}{V} = \frac{\frac{W_o}{S_b} - \frac{W_o}{S_t}}{\frac{W_o}{S_b}} = \frac{S_t - S_b}{S_t} = \frac{1 - S_b}{S_t}$$

where

W_o = oven-dry weight of aggregate,

S_b = bulk specific gravity,

S_t = true specific gravity.

Table 30

Sample Data Sheet for Linear Traverse Studies
of Polished Sections of Cherts

Sample No. 2077 2.45 minus Date June 2, 1960

Polished Section No. #3

Traverse Number	Solids (mm.)	Voids (mm.)	Totals (mm.)
1	10.573	0.655	11.228
2	10.247	0.945	11.192
3	10.380	0.870	11.250
4	10.405	0.945	11.350
5	10.506	0.870	11.376
6	10.700	0.760	11.460
7	10.268	0.768	11.036
8	10.050	0.865	10.915
9	10.010	0.721	10.731
10	9.818	0.895	10.713
Totals	102.957	8.294	111.251

$$\text{Per cent voids} > 5 \text{ microns in diameter} = \left(\frac{\Sigma \text{ voids}}{\Sigma \text{ solids} + \Sigma \text{ voids}} \right) \times 100$$

$$= \left(\frac{8.294}{111.251} \right) \times 100 = 7.46\%$$

Sample Calculations for Specific Surface Areas and Spacing Factors of Air Voids in Concrete Beams

By means of the linear traverse technique it was found that the concrete in beam S6-3 contained 3.32 per cent air voids and had an average of 4.20 voids per inch of traverse. The calculations of specific surface area and spacing factor for the voids were as follows (40):

Specific surface area of voids,

$$\alpha = \frac{4n}{A} = \frac{(4)(4.20)}{(.0332)} = 506 \text{ sq.in./cu. in.}$$

Hypothetical number of voids per cu. in.,

$$N = \frac{A\alpha^3}{36\pi} = \frac{(0332)(506)^3}{36\pi} = 38,000$$

Paste content per unit volume of mix,

$p = .126$ (from mix design)

Radius of hypothetical sphere,

$$r_h = \frac{3}{\alpha} = \frac{3}{506} = .00593 \text{ in.}$$

Radius of "sphere of influence,"

$$r_m = \frac{\sqrt{3}}{2}\left(\frac{p + A}{N}\right)^{1/3} = \frac{\sqrt{3}}{2}\left(\frac{.126 + .0332}{38,000}\right)^{1/3}$$

$$= .01397 \text{ in.}$$

Spacing factor,

$$L = r_m - r_h = (.01397 - .00593) = 0.008 \text{ in.}$$

VITA

VITA

Robert Lee Schuster was born on August 29, 1927, in Chehalis, Washington. He attended the elementary and high schools of that city and graduated from Chehalis High School in June, 1945.

Mr. Schuster entered the State College of Washington in September, 1945, and received the Bachelor of Science degree in geology from that institution in January, 1950. After graduation, he worked for six months as an engineering geologist for the Materials Testing Laboratory of the Washington State Department of Highways.

In September, 1950, Mr. Schuster began graduate study and employment as a graduate teaching assistant in geology at Ohio State University. He received his Master of Science degree in geology from this university in June, 1952.

During the summers of 1951 and 1952, Mr. Schuster was employed as a field geologist in Alaska by the American Geographical Society. From October, 1952, to January, 1955, he conducted geologic field studies in Alaska, Canada, and Greenland as an engineering geologist with the Snow, Ice, and Permafrost Research Establishment, Corps of Engineers, U. S. Army.

In September, 1955, Mr. Schuster entered Purdue University and began graduate study in civil engineering. During the 1955-56 academic year, he served as a graduate teaching assistant in the School of Civil Engineering. In February, 1957, he was appointed to

the full-time staff, serving as half-time instructor in engineering geology and half-time research assistant (later research engineer) for the Joint Highway Research Project. He presently is employed in this capacity. In June, 1958, Mr. Schuster received the Master of Science in Civil Engineering degree from Purdue University.

Mr. Schuster is a member of the American Society of Civil Engineers, the Geological Society of America, the Indiana Academy of Science, Sigma Xi, Tau Beta Pi, and Sigma Gamma Epsilon. He also is a registered Professional Engineer in the State of Indiana.

His publications include the following which are directly related to study of concrete aggregates:

Schuster, R. L., "Sand and Gravel Resources of Indiana (abst.)," Bulletin, Geological Society of America, Vol. 68, No. 12, p. 1900, 1957.

Schuster, R. L., "A Review of Research on Deleterious Substances in Concrete Aggregates," Technical Paper, Joint Highway Research Project, Purdue University, No. 37, 1957.

Woods, K. B., McLaughlin, J. F., and Schuster, R. L., "Quality Aggregates for Indiana Highways," Engineering Bulletin, Purdue University, Vol. 42, No. 4, pp. 81-94, 1958.